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Quality Assurance Handbook for Air Pollution Measurement Systems Volume II. Ambient Air Specific Methods

Environmental Monitoring & Support Lab, Research Triangle Pk, NC

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QUALITY ASSURANCE HANDBOOK FOR AIR POLLUTION MEASUREMENT SYSTEMS

Volume II — Ambient Air Specific Methods

U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Environmental Monitoring and Support Laboratory
Research Triangle Park, North Carolina 27711

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16. ABSTRACT

This Handbook includes quality assurance guidelines on ambient air measurement systems. Regardless of the scope and magnitude of the ambient air measurement system, there are a number of common considerations pertinent to the production of quality data. These common parameters are discussed in Section 2.0 of Volume II and include quality assurance guidelines in the areas of: 1. Sampling site selection considerations, 2. Sampling considerations, 3. Data handling and reporting, 4. Compliance monitoring reference and equivalent methods, and 5. Recommended minimum quality assurance program for ambient air monitoring systems. The remainder of Volume II contains pollutant-specific quality assurance guidelines. Initially Volume II includes guidelines for the following pollutant-specific measurement systems: Section 2.1 reference method for determination of sulfur dioxide (pararosaniline method) and Section 2.2 reference method for determination of suspended particulates (high volume method). Operators and managers responsible for ambient air measurements will find Volume II useful in planning quality assurance.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Research Triangle Park, North Carolina 27711

QUALITY ASSURANCE HANDBOOK FOR AIR POLLUTION MEASUREMENT SYSTEMS

VOLUME II - AMBIENT AIR SPECIFIC METHODS

The U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory (EMSL), located at the Research Triangle Park, North Carolina, is responsible for providing guidelines on quality assurance program development to organizations making air pollution measurements. Enclosed is a copy of the "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II - Ambient Air Specific Methods," which is the second of a three volume series of resource documents for quality assurance for air pollution measurement systems. This volume contains quality assurance guidelines on ambient air pollutant measurement systems. Regardless of the scope and magnitude of the measurement system, there are a number of common considerations pertinent to the production of quality data. These common parameters are discussed in Section 2.0. The remainder of Volume II contains quality assurance guidelines for specific ambient air pollutant measurement methods. The first two methods in Volume II are the reference methods for the determination of sulfur dioxide (Section 2.1) and suspended particulates (Section 2.2) in the atmosphere. Some of the other sections currently under development include quality assurance guidelines for continuous monitoring for nitrogen dioxide by chemiluminescence, measurement of nitrogen dioxide by sodium arsenite, and continuous monitoring for sulfur dioxide by flame photometric detection.

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PURPOSE AND OVERVIEW OF THE QUALITY ASSURANCE HANDBOOK

The purpose of this Quality Assurance Handbook for Air Pollution Measurement Systems is to provide guidelines and procedures for achieving quality assurance in air pollution measurement systems. It is intended to serve as a resource document for the design of quality assurance programs and to provide detailed method descriptions for certain measurement processes that can be used directly in implementing the quality assurance program. This Handbook should be particularly beneficial to operators, project officers, and program managers responsible for implementing, designing and coordinating air pollution monitoring projects.

Volume I - Principles

Volume I contains brief discussions of the elements of quality assurance. Expanded discussions of technical points and sample calculations are included in the Appendices. The discussion of each element, therefore, is structured to be brief and at the same time to highlight its most important features. Organizations developing and implementing their own quality assurance plans will find Volume I useful for general guidance.

Volume II - Ambient Air Specific Methods

Volume II contains quality assurance guidelines on ambient air measurement systems. Regardless of the scope and magnitude of ambient air measurement systems, there are a number of common considerations pertinent to the production of quality data. These common parameters are discussed in Section 2.0 of Volume II and include quality assurance guidelines in the areas of:

1. Sampling site selection considerations - network design, representative sampling consistent with objectives, meteorological and topographical restraints, and sampling schedules.

2. Sampling considerations - environmental controls, probe and manifold design, and support services.

3. Data handling and reporting considerations - data recording systems, data validation, and systematic data management.

4. Compliance monitoring - reference and equivalent methods.

5. Recommended quality assurance program for ambient air measurements.

The remainder of Volume II contains method and/or principle description and quality assurance guidelines for specific pollutants. Each pollutant-specific section contains the following information.

1. Procedures for procurement of equipment and supplies.
2. Calibration procedures.
3. Step-by-step description of sampling, reagent preparation, and analysis procedures, as appropriate, depending upon the method or principle in the case of equivalencies.
4. Method of calculation and data processing checks.
5. Preventive maintenance procedures.
6. Recommended auditing procedure - audits to be performed during the sampling, analysis, and data processing.
7. Recommended standards for establishing traceability.
8. Pertinent references.
9. Blank data forms for convenience of the Handbook user (data forms are partially filled in within the main text for illustration purposes).

Method activity matrices are given at the end of appropriat subsections for the purpose of summarizing the quality assurance functions therein. The matrix includes the activity, acceptance limits, method and frequency of the quality assurance checks, and recommended action if the acceptance limits are not satisfied.

Volume II contains two pollutant-specific measurement systems. These are:

1. Reference Method for the Determination of Sulfur Dioxide in the Atmosphere (Pararosaniline Method).

2. Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method).

As additional methods are added to Volume II, these will be sent to Handbook users through the document control system as described in Section 1.4.1 of Volume I of this Handbook.

Volume III - Stationary Source Specific Methods

Volume III contains quality assurance guidelines on stationary source specific methods. The format for Volume III is patterned after that of Volume II.

Regardless of the scope and purpose of the emissions testing planned, there are a number of common considerations pertinent to the production of quality data. These common parameters are discussed in Section 3.0 of Volume III and include quality assurance guidelines in the area of:

1. Planning the test program.
2. General factors involved in source testing.
3. Data evaluation.

The remainder of Volume III contains quality assurance guidelines for specific measurement methods. The measurement systems to be included in Volume III will be:

Method 2 - Determination of Stack Gas Velocity and Volumetric Flow Rate (Type-S Pitot Tube).

Method 3 - Gas Analysis for Carbon Dioxide, Oxygen, Excess Air, and Dry Molecular Weight.

Method 4 - Determination of Moisture in Stack Gases.

Method 5 - Determination of Particulate Emissions from Stationary Sources.

Method 6 - Determination of Sulfur Dioxide Emissions from Stationary Sources.

Method 7 - Determination of Nitrogen Oxide Emissions from Stationary Sources.

Method 8 - Determination of Sulfuric Acid Mist and Sulfur Dioxide from Stationary Sources.

Method 9 - Visual Determination of Opacity Emissions from Stationary Sources.

Method 10 - Reference Method for the Determination of CO Emissions from Stationary Sources by NDIR Spectrometry.

As additional methods are added to Volume III, these will be sent to Handbook users through the document control system used for the Handbook.

SECTION 2.0

COMMON QUALITY ASSURANCE GUIDELINES FOR AMBIENT AIR MONITORING SYSTEMS

OUTLINE

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SUMMARY

Regardless of the scope and magnitude of air monitoring systems, there are a number of common considerations pertinent to the production of good quality data. These common parameters include quality assurance guidelines in the areas of:

1. Selection of the sampling site.
2. Control of physical sampling parameters.
3. Data handling and reporting.
4. Compliance monitoring - reference and equivalent methods.
5. Recommended quality assurance program for ambient air measurements.

If guidelines for monitoring applications included in these five categories are not followed, the result may be the collection and storage of data that are non-representative of ambient conditions. While not traditionally considered to be elements of a quality assurance program, these parameters directly influence the overall validity of the collected data and therefore are included as a necessary adjunct of any quality assurance program for ambient air monitoring.

1.0 SAMPLING SITE SELECTION CONSIDERATIONS

The need for an air quality monitoring program usually is related to one or more of the following objectives:

1. To judge compliance with and/or progress made toward meeting ambient air quality standards.
2. To activate emergency control procedures that prevent or alleviate air pollution episodes.
3. To observe pollution trends throughout a region, including nonurban areas.
4. To provide a data base for research evaluation of effects; urban, land use, and transportation planning; development and evaluation of abatement strategies; and development and validation of diffusion models.

The monitoring objectives by pollutant are given in Table I-3 of reference 1.

Sampling site and equipment requirements are generally divided into three categories, consistent with desired averaging times:

1. Continuous--Pollutant concentrations determined with automated methods and recorded or displayed continuously.
2. Intermittent--Pollutant concentrations determined with manual or automated methods from integrated hourly or daily samples on a fixed schedule.
3. Static--Pollutant estimates or effects determined from longer-term (weekly or monthly) exposure of qualitative measurement devices or materials.

Air quality monitoring sites that employ automatic equipment to continually sample and analyze pollutant levels may be classified as primary. Primary monitoring stations are generally located in areas where pollutant concentrations are expected to be among the highest and in areas of highest population density and, as such, are often employed in health effects research networks. In addition, these stations are designed as a part of the air pollution episode warning system.

The quality assurance plan[a] should include specific documentation of site characteristics for each monitoring station employed. This information will assist in providing objective input into the evaluation of data generated at that site. Typically, the site identification record should include:[2]

1. Data acquisition objective
   (Air quality standards monitoring)
2. Station type
   (Stationary, mobile, etc.)
3. Instrumentation checklist
   (Manufacturer model no., pollutant measurement technique, etc.)
4. Sampling system
   (Probe type, height, flow, etc.)
5. Location of the station
   (Site category, i.e., urban/industrial, suburban/commercial, etc.; and physical location, i.e., address, AQCR, UTM coordinates, etc.)
6. Influential pollutant sources
   (Point and area sources, proximity, pollutant density, etc.)
7. Topography
   (Hills, valleys, bodies of water, trees - type and size, proximity, orientation, etc., picture of a 360° view from the probe of the monitoring site)
8. Atmospheric exposure
   (Unrestricted, interferences, etc.)
9. Site diagram
   (Sample flowsheet, service lines, equipment configuration, etc.)

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[a] Minimum suggested content for a quality assurance plan is discussed in the Quality Assurance Handbook for Air Pollution Measurement Systems, Vol. I, in Section 1.4.23 entitled "Quality Assurance Plans for Projects and Programs".

1.1 Network Design Considerations

In designing an air quality monitoring program, the following four criteria for locating sites should be considered, either singly or in combination, depending upon the objective of sampling:

1. Orient monitoring sites to measure the impacts of known pollutant emission categories on air quality.
2. Orient monitoring sites relative to population density to measure receptor-dose levels, both short and long-term.
3. Orient monitoring sites to measure the impacts of known pollutant emission sources (area and point) on air quality.
4. Orient monitoring sites to obtain measurements representative of areawide air quality.

In order to select locations according to these criteria, it is necessary to have detailed information of the location of sources of emission, the geographical variability of ambient pollutant concentrations, meteorological conditions, and population density.

Therefore, the selection of the number, location, and type of sampling stations is a complex problem without a purely objective solution. The variability of sources and their intensity, terrain, meteorological conditions, and demographic features requires that each network be developed individually. The network chosen will be the result of subjective judgments, based upon available evidence and the experience of the decision team.

The sampling site selection process involves consideration of economic, logistic, atmospheric, and pollutant reaction factors in addition to the motivation for and the objective of the sampling program. None of the factors stand alone; each is dependent in part upon the others. However, the objective of the sampling program must be clearly defined before the selection process can be initiated. The initial definition of priorities may have to be reevaluated after consideration of the remaining factors before the final locations are chosen.

The economic considerations are rather clearly defined. The amount of money required for data gathering (instrumentation, installation, maintenance, data retrieval system), data analysis, quality assurance, and data interpretation must be balanced against the available monies (current and projected) and the cost-benefits of additional or relocated sampling sites.

The logistical problems involve the means of obtaining, analyzing, and interpreting the data. It must be determined whether the current staff can manage the proposed system, or more or fewer persons are required to accomplish the tasks. It is important to decide if the information derived is necessary on a real-time basis or if several weeks may elapse before it is needed.

The atmospheric problems pertain to definition of the spatial and temporal variability of the pollutants and their transport. Local effects upon the air trajectories by buildings, terrain, and heat sources or sinks can produce local anomalies of excessive pollutant concentration. Wind velocity, wind shear, and atmospheric stability greatly influence the dispersal of pollutants.

Additionally, a sampling site or an array of sites for one pollutant may be inappropriate for another pollutant species because of the configuration of sources, local meteorology, or terrain. Atmospheric chemical reactions such as the production of ozone in the presence of nitrogen oxides and hydrocarbons, and the time delay between nitrogen oxides and hydrocarbons emissions and the peak ozone values may require either a sampling network for the precursors of ozone and/or a different network for the actual ozone measurement. Pollutants undergo changes in their composition between their emission and their detection; therefore, the impact of that change upon the measuring system should be considered.

While the interaction of the factors identified in the above paragraphs is complex, the siting problem can be resolved. It requires experience in the operation of air quality measurement systems, estimates of air quality, field and theoretical studies of atmospheric diffusion, and consideration of atmospheric chemistry and air pollution effects; this combined expertise provides the input for determination of optimum sampling site selection in order to obtain data representative of monitoring objectives.

1.2 Representative Sampling

Assuring the collection of a representative air quality sample depends on the following factors:

1. Locating the sampling site and determining network size consistent with monitoring objectives.
2. Restraints on the sampling site imposed by meteorology.
3. Restraints on the sampling site imposed by local topography, emission sources, and physical constraints.
4. Sampling schedules consistent with monitoring objectives.

1.2.1 Locate Sampling Site and Determine Network Size - Consistent with monitoring objectives previously noted, networks are designed to meet at least one of four major objectives. The following tabulation presents examples of currently implemented networks applicable to each of these "objectives" categories:

Objective	Network	Comment
Compliance monitoring	SIP (State Implementation Plan)	To demonstrate attainment or maintenance of Air Quality Standards
Emergency episode monitoring	SIP/local agency emergency control program	To activate immediate, short-term, emission controls for prevention of episodes
Trend monitoring	NASN (National Air Sampling Networks)	To fulfill mandate of Federal legislation
Research monitoring	CHAMP (Community Health Air Monitoring Program)	To determine long-term pollutant trend in selected areas with respect to health effects

Compliance Monitoring - The information required for selecting sampler location is essentially the same as that for determining the number of samplers, i.e., isopleth maps, population density maps, and source locations. Following are suggested guidelines:

1. The priority area is the zone of highest pollutant concentration within the region. One or more stations are to be located in this area.

2. Close attention should be given to densely populated areas within the region, especially when they are in the vicinity of heavy pollution.

3. For assessing the quality of air entering the region, stations must also be situated on the periphery of the region. Meteorological factors such as frequencies of wind direction are of primary importance in locating these stations.

4. For determining the effects of future development on the environment, sampling should be undertaken in areas of projected growth.

5. A major objective of surveillance is evaluation of progress made in attaining the desired air quality. For this purpose, sampling stations should be strategically situated to facilitate evaluation of the implemented control tactics.

6. Some information of air quality should be available to represent all portions of the regions.

Some stations will be capable of fulfilling more than one of the functions indicated; e.g., a station located in a densely populated area can indicate population exposures and also document the changes in pollutant concentrations resulting from control strategies employed in the area.

Table 1.1 presents general guidelines for distribution of mechanical and automatic sampling stations. The basis for the number of monitoring sites required in a State Implementation Plan network is the Code of Federal Regulations.[3] In practice, siting determinations are also affected strongly by such factors as location of major polluting sources, characteristics of individual pollutants, and local meteorology.

Emergency Episode Monitoring - For episode avoidance purposes, data are needed quickly--in no less than a few hours after the sensor is contacted by the pollutant. While it is possible to obtain data rapidly by on-site manual data reduction and telephone reporting, there is a trend toward automated monitoring networks. Obviously, the severity of the problem, size of the receptor area, and availability of resources influence both the scope and sophistication of the system.

It is necessary to utilize continuous air samplers because an episode lasts only a few days and the control actions taken must be based on "real-time" measurements correlated with the decision criteria. Based on alert criteria now in use, 1-hour averaging times are adequate for surveillance of episode conditions. Shorter averaging times provide information on data-collecting excursions but increase the need for automation

Table 1.1 REGULATORY MINIMUM NUMBER OF MONITORING SITES[a]

Classification of region	Pollutant	Measurement method or principle [i]	Minimum frequency of sampling	Region population	Minimum number of air quality monitoring sites [c]
I	Suspended particulates	High volume sampler	One 24-hour sample every 6 days [a].	Less than 100,000	4.
				100,000-1,000,000	4+0.6 per 100,000 population.[c]
				1,000,001-5,000,000	7.5+0.25 per 100,000 population.[c][h]
				Above 5,000,000	12+0.16 per 100,000 population.
		Tape sampler	One sample every 2 hours		One per 250,000 population [c] up to eight sites.
	Sulfur dioxide	Pararosaniline or equivalent [d].	One 24-hour sample every 6 days (gas bubbler).[a]	Less than 100,000	3.
				100,000-1,000,000	2.5+0.5 per 100,000 population.[c]
				1,000,001-5,000,000	6+0.15 per 100,000 population.[c][h]
				Above 5,000,000	11+0.05 per 100,000 population.
			Continuous	Less than 100,000	1.
				100,000-5,000,000	1+0.15 per 100,000 population.[c]
				Above 5,000,000	6+0.05 per 100,000 population.[c]
	Carbon monoxide	Nondispersive infrared or equivalent.[e]	Continuous	Less than 100,000	1.
				100,000-5,000,000	1+0.15 per 100,000 population.[c]
				Above 5,000,000	6+0.05 per 100,000 population.[c]
	Photochemical oxidants	Gas phase chemiluminescence or equivalent.[f]	Continuous	Less than 100,000	1.
				100,000-5,000,000	1+0.15 per 100,000 population.[c]
				Above 5,000,000	6+0.05 per 100,000 population.[c]
	Nitrogen dioxide	24-hour sampling method (Jacobs-Hochheiser method).	One 24-hour sample every 14 days (gas bubbler).[b]	Less than 100,000	3.
				100,000-1,000,000	4+0.6 per 100,000 population.[c]
				Above 1,000,000	10.
II	Suspended particulates	High volume sampler	One 24-hour sample every 6 days [a].		3.
		Tape sampler	One sample every 2 hours.		1.
	Sulfur dioxide	Pararosaniline or equivalent [d].	One 24-hour sample every 6 days (gas bubbler).[a]		3.
			Continuous		1.
III [g]	Suspended particulates	High volume sampler	One 24-hour sample every 6 days [a].		1.
	Sulfur dioxide	Pararosaniline or equivalent [d].	One 24-hour sample every 6 days (gas bubbler).[a]		1.

[a] Equivalent to 61 random samples per year.

[b] Equivalent to 26 random samples per year.

[c] Total population of a region. When required number of samplers includes a fraction, round-off to nearest whole number.

[d-f] [Reserved]

[g] It is assumed that the Federal motor vehicle emission standards will achieve and maintain the national standards for carbon monoxide, nitrogen dioxide, and photochemical oxidants; therefore, no monitoring sites are required for these pollutants.

[h] In interstate regions, the number of sites required should be prorated to each State on a population basis.

[i] Named methods and principles, except the tape sampler method, are described in Part 50 of this chapter. The tape sampler method is described in Hemeon, W. C. L., Haines, G. F., Jr. and Ide, H. M., "Determination of Haze and Smoke Concentrations by Filter Paper Samplers", J. Air Pollution Control Association, Vol. 3, pp. 22-28, 1953. Use of these and other methods shall be as specified in § 51.17a.

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[a] CFR, Part 51.17, Air Quality Surveillance, July 1975, page 43.

because of the bulk of the data obtained. Averaging times longer than six hours are not desireable because of the delay in response this imposes. After an alert is announced, data are needed quickly so that requests for information on the event can be met.

Collection and analysis must be accomplished rapidly if the data are to be useful immediately. There is no time to check out the methods, run blanks, calibrate, etc., after the onset of episode conditions. In order for the instrument to be maintained in peak operating condition, personnel must be stationed at the sites during the episode or automated equipment must be operated that can provide automatic data transmission to a central location.

The instruments employed for measuring the pollutant parameters utilize wet chemical techniques or physical methods. Chemical analysis always involves the use of consumable supplies. The chemicals must be replaced on a schedule that is consistent with their stability and with the rate at which samples are taken. Currently used instruments require adequate supplies of chemicals for operation for a period of three months in order that the supplier can comply with delivery schedules. In some cases, analytical reagents for specific air contaminants deteriorate rapidly and should have protective storage. Physical methods of measurement are performed with relatively complex equipment. These instruments must be installed correctly and cared for by trained personnel. Their accuracy is affected by mechanical shock, ambient temperature extremes, voltage supply stability, dirty or dusty atmospheres, and corrosive chemicals.

Episode conditions threaten human welfare, and monitoring sites should be located in areas where this welfare is most threatened:

1. In densely populated areas.
2. Near large stationary sources of pollutants.
3. Near hospitals.

4. Near high-density traffic interchanges.
5. In homes for the aged.

A network of sites is useful in determining the range of pollutant concentrations within an area. Although the most desirable monitoring sites are not necessarily the most convenient, consideration should be given, for reasons of access, security, and existing communications, to the use of public buildings: schools, firehouses, police stations, hospitals, and water or sewage plants.

Trend monitoring - As typified by the National Air Surveillance Network (NASN), trend monitoring is characterized by locating a minimal number of monitoring sites across as large an area as possible. The program objective is to determine, in a broad sense, the extent and nature of air pollution as well as determine the variation in the measured levels of atmospheric contaminants in respect to geographic, socioeconomic, climatologic and other factors. The data acquired are useful in planning epidemiological investigations and also provide the background against which more intensive community and statewide studies of air pollution can be conducted.

Urban sampling stations are usually located in the most densely populated areas of a region. In most regions there are several urban sites.

Nonurban station locations include various topographical categories such as farmland, desert, forest, mountain, and coastal. The nonurban stations are not specifically selected to be "clean air" control sites for urban areas, but they do provide for a relative comparison between some urban and nearby nonurban areas.

In interpreting trend data one must consider the limitations imposed by the network design. Even though precautions are taken to ensure that each sampling site is as representative as possible of the designated area, it is impossible to be totally certain that the measurements obtained at a specific site are not sometimes unduly influenced by local factors. Such factors might include topography, structures, and sources of pollution in the immediate vicinity of the site, and other variables, the effect of which cannot always be accurately anticipated but which should be considered in network design. It must be kept in mind that when comparisons are made among pollution levels for various areas, they are valid only insofar as the sites are comparable.

Research Monitoring - An example of a research-oriented air quality monitoring effort is the EPA's Community Health Air Monitoring Program (CHAMP), which is providing data to develop criteria for both short- and long-term air quality standards. Air monitoring networks related to health effects are composed of integrating samplers for both determining pollutant concentrations for 24 hours or longer for developing long-term ($\geq$ 24 hours) ambient air quality standards. These studies require that monitoring points be located so that the resulting data represent the population group under study. The monitoring stations are therefore established in the center of small, well-defined residential areas within a community. Data correlations are made between observed health effects and observed air quality exposure.

Requirements for aerometric monitoring in support of health studies are:

1. Station must be located in or near population under study.

2. Pollutant sampling averaging times must be sufficiently short to allow for use in acute health effects studies that form the scientific basis for short-term standards.

3. Sampling frequency should be sufficient to characterize air quality as a function of time, usually daily.

4. System should be flexible and responsive to emergency conditions with data available on short notice.

Figure 1.1 graphically represents CHAMP, a prime example of a research monitoring network.

1.2.2 Meteorological Factors that Affect Representative Sample Collection - Meteorology must be considered in determining not only the geographical location of a monitoring site, but also such factors as height, direction and extension of sampling probes. Meteorological parameters having the greatest influence on dispersion of pollutants are the direction, speed, and variation of wind.

Wind direction provides an indication of the general movement of pollutants in the atmosphere. Review of available data can indicate mean wind direction in the vicinity of the major sources of emissions.

The effects of wind speed are two-fold. First, wind speed determines the travel time from source to receptor. Second, wind speed affects dilution in the downwind direction, i.e., concentration of air pollutants is inversely proportional to wind speed. Turner[4] gives an example:

> If a continuous source is emitting a certain pollutant at a rate of 10 grams/second, and the wind speed is 1 meter/second, then in a downwind length of the plume of 1 meter will be contained 10 grams of the pollutant, since one meter of air moves past the source each second. Next, consider that the conditions of emissions are the same, but the wind speed is 5 meters/second. In this case since 5 meters of air moves past the source each second, each meter of plume length contains 2 grams of pollutant.

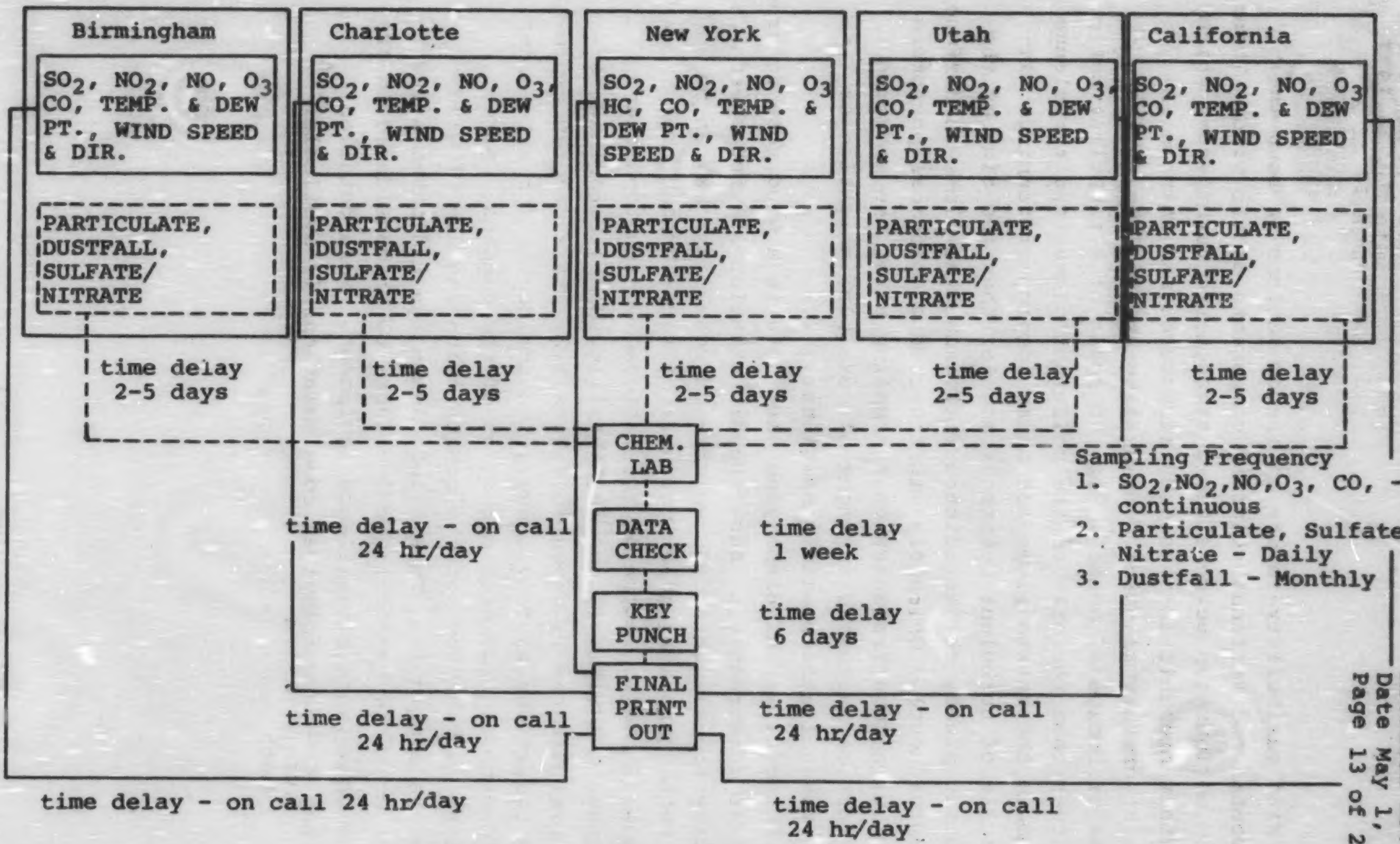

Birmingham
SO_2, NO_2, NO, O_3 CO, TEMP. & DEW PT., WIND SPEED & DIR.
PARTICULATE, DUSTFALL, SULFATE/ NITRATE
Charlotte
SO_2, NO_2, NO, O_3, CO, TEMP. & DEW PT., WIND SPEED & DIR.
PARTICULATE, DUSTFALL, SULFATE/ NITRATE
New York
SO_2, NO_2, NO, O_3 HC, CO, TEMP. & DEW PT., WIND SPEED & DIR.
PARTICULATE, DUSTFALL, SULFATE/ NITRATE
Utah
SO_2, NO_2, NO, O_3, CO, TEMP. & DEW PT., WIND SPEED & DIR.
PARTICULATE, DUSTFALL, SULFATE/ NITRATE
California
SO_2, NO_2, NO, O_3 CO, TEMP. & DEW PT., WIND SPEED & DIR.
PARTICULATE, DUSTFALL, SULFATE/ NITRATE
time delay 2-5 days
time delay 2-5 days
time delay 2-5 days
time delay 2-5 days
time delay 2-5 days
CHEM. LAB
DATA CHECK
KEY PUNCH
FINAL PRINT OUT
time delay - on call 24 hr/day
time delay 1 week
time delay 6 days
time delay - on call 24 hr/day
time delay - on call 24 hr/day
time delay - on call 24 hr/day
time delay - on call 24 hr/day
Sampling Frequency
1. SO_2,NO_2,NO,O_3, CO, - continuous
2. Particulate, Sulfate/ Nitrate - Daily
3. Dustfall - Monthly

Figure 1.1 CHAMP air monitoring program

Wind variability refers to random motions in both the horizontal and vertical velocity components of the wind. These random motions can be considered atmospheric turbulence, which is either mechanical (caused by structures and changes in terrain) or thermal (caused by heating and cooling of land masses or bodies of water). If the scale of a turbulent motion is larger than the size of the pollutant plume, the turbulence will move the entire plume and cause looping or fanning. If the scale of turbulent motion is smaller than the size of the pollutant plume, the turbulence will cause the plume to diffuse (or spread out). Obviously, if these meteorological phenomena impact with some frequency on a sampling site, the measured data must be evaluated in light of possible fumigation or other unusual atmospheric conditions.

A useful way of displaying wind data is a wind rose diagram, which is constructed to show the distribution of wind speeds and directions. Data from which wind rose diagrams can be constructed are available in tabulated form through the National Climatic Center of the National Oceanographic and Atmospheric Administration in Asheville, North Carolina. A wind rose diagram is shown in Figure 1.2. This diagram represents conditions as they converge on the center (or site under consideration) from each direction of the compass. More complete explanations of the construction and use of wind roses are available. This brief discussion indicates a few of the possible meteorological effects to be considered in the siting of network surveillance equipment. More detailed guidance for meteorological considerations is available.[1,4]

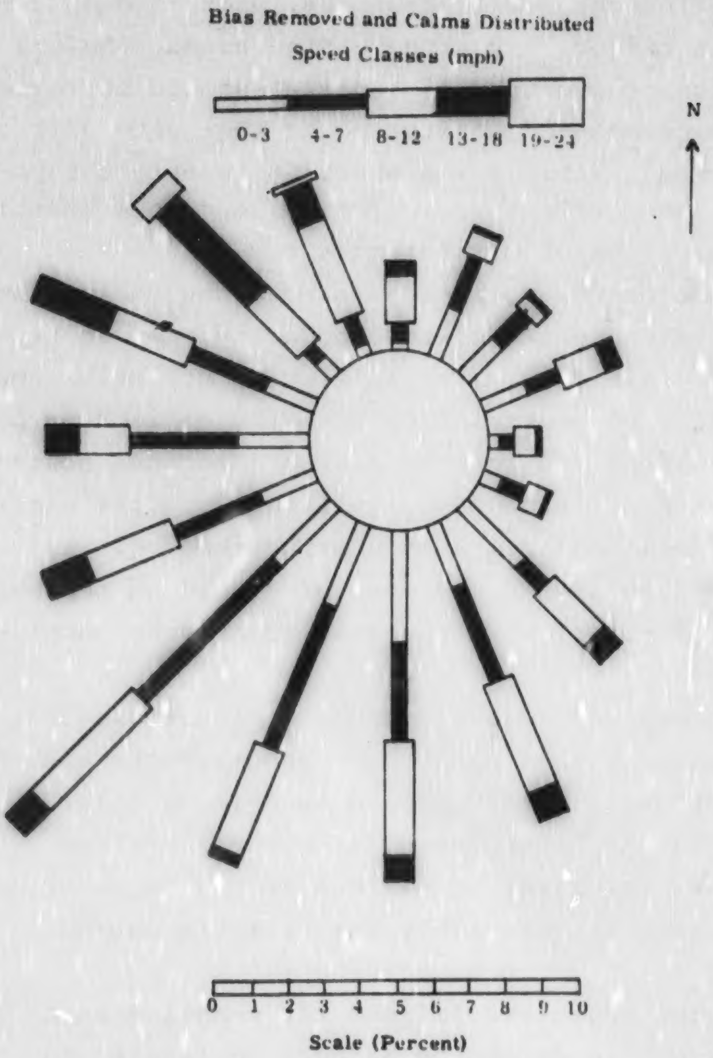


Figure 1.2 Wind rose pattern.

1.2.3 Topographical Features that Affect Representative Sample Collection - The transport and diffusion of air pollutants is complicated by topographic features. Minor topographic features may exert small influence; major features, such as deep river valleys or mountain ranges, may affect large areas. Before final site selection, topography of the area should be reviewed to ensure that the purpose of monitoring at that site will not be adversely affected. Table 1.2 summarizes important topographic features, their effects on air flow, and some examples of influence on selection of monitoring sites.

Final placement of the monitor at a selected monitoring site depends on physical obstructions and activities in the immediate area, accessibility, availability of utilities and other support facilities, correlation with the defined purpose of the specific monitor, and monitor design. Because obstructions such as trees and fences can significantly alter air flow, monitors should be removed from such obstructions. It is important that air flow around the monitor should be representative of the general air flow in the area to prevent sampling bias.

Network designers should avoid sampling locations that are unduly influenced by down-wash or by ground dust, such as a rooftop air inlet near a stack or a ground-level inlet near an unpaved road. In the latter case, either elevate the sampler intake above the level of maximum ground turbulence effect or simply place it reasonably far from the source of ground dust.

Depending on the objective defined for sampling at a particular site, i.e., to determine background levels, to determine maximum concentrations, etc., the monitors are placed according to exposure to pollution, including exposure to motor vehicle emissions. Therefore, in most every instance, a practical consideration of unavoidable physical restraints to

Table 1.2 RELATIONSHIPS OF TOPOGRAPHY, AIR FLOW, AND MONITORING SITE SELECTION

Topographic feature	Influence on air flow	Influence on monitoring site selection
1. Slope	Downward air currents at night and on cold days; upslope winds on clear days when valley heating occurs.	Slopes and valleys are generally special sites for air monitors because pollutants are generally not well dispersed. Concentration levels would not be representative of other geographic areas. A possible application would be placement of a monitor to determine concentration levels in a population or industrial center in the valley.
2. Valley	Slope winds and valley channelled winds. General tendency toward down-slope and down-valley winds. Tendency toward inversions.	
3. Water	Sea or lake breezes inland or parallel to shoreline during the day or in cold weather. Land breezes at night.	Monitors placed on shorelines could be useful for background readings or for obtaining pollution data on water traffic.
4. Hill	Sharp ridges cause turbulence. Flow is around obstructions during stable conditions; flow is over obstructions during unstable conditions.	Depends on source orientation. Since source emissions upwind may be mixed down the slope, siting at the foot of the hill may not be advantageous. Source emissions downwind may be downwashed near the source. Monitoring close to a source may be desirable if population centers are adjacent or if monitoring is done to protect workers.
5. Natural or man-made obstruction	Eddy effects	Placement near obstructions may not give representative readings.

optimum representative sample collection must be made. This consideration should include categorization of sites relative to their localized placement. Suggested categories relating to sampling site placement for measuring a corresponding pollutant impact are as follows:

Category A. Ground Level Station. Heavy pollutant concentrations--high potential for pollutant buildup. Site 3 to 5 meters (10-16 feet) from major traffic artery with local terrain features restricting ventilation. Sampler probe 3 to 6 meters (10-20 feet) above ground.

Category B. Ground Level Station. Heavy pollutant concentrations--minimal potential for pollutant buildup. Site 3 to 15 meters (15-50 feet) from major traffic artery with good natural ventilation. Sampler probe 3 to 6 meters (10-20 feet) above ground.

Category C. Ground Level Station. Moderate pollutant concentrations. Site 15 to 60 meters (50-200 feet) from major traffic artery. Sampler probe 3 to 6 meters (10-20 feet) above ground.

Category D. Ground Level Station. Low pollutant concentrations. Site 60 meters (200 feet) or more from traffic artery. Sampler probe 3 to 6 meters (10-20 feet) above ground.

Category E. Air Mass Station. Sampler probe is between 6 and 45 meters (20 and 150 feet) above ground. Two sub-classes: (1) good exposure from all sides (e.g., on top of building), and (2) directionally biased exposure (probe extended from window).

Category F. Source Oriented Station. Sampler is adjacent to point source; monitoring yields data directly relatable to source of emission.

Guidelines for placing air monitoring instruments in areas of estimated maximum pollutant concentrations have been developed as given in Table 1.3. Guidelines are different for stations defining average 1-hour CO concentrations and for stations defining average 8-hour CO concentrations because people are not ordinarily exposed to CO concentrations over a period of 8 hours in densely trafficked downtown areas. When only one sampling site must satisfy the minimum surveillance requirement of a monitoring plan, that site must meet the guidelines for averaging over 8 hours. Distance from the street is specified in the location guidelines for stations monitoring for carbon monoxide because streets are the primary locations of CO emissions. For the same reason, height of the air inlet from the ground is more restrictive in monitoring for CO than for other pollutants.[5]

1.2.4 Sampling Schedules Consistent with Monitoring Objectives - Current Federal regulations specify the frequency of sampling for criteria pollutants to meet minimum SIP surveillance requirements. Continuous sampling is specified except for 24-hour measurements of total suspended particulate matter and 24-hour integrated values of SO_2 and NO_2. The high-volume and gas impinger measurements are required at least once every six days, equivalent to about 61 random samples per year. Twenty-four-hour samples should be taken from midnight (local standard time) to midnight and thus represent calendar days to permit direct utilization of the sampling data with standard daily meteorological summaries.

The following are recommended frequencies for noncontinuous high-volume and impinger sampling in order to adequately define TSP, SO_2, and NO_2 levels:

Table 1.3 SUMMARY OF GUIDELINES FOR STATION SITING AND PROBE PLACEMENT*

		Site Selection	Probe Placement (Meters)		
Pollutant	Site Type	Site Location	Height Above Ground	Vertical Clearance[a]	Horizontal Clearance[b]
Sulfur dioxide	Peak	Maximum point determined from atmosphere diffusion model, historical data, emission density, and representative of population exposure.	3 to 15	1 to 2	> 2
	Neighborhood	Determined on basis of population patterns and air quality gradients	3 to 15	1 to 2	> 2
	Background	Nonurban area within Region	3 to 15	1 to 2	> 2
Suspended Particulates	Peak	Same as for SO_2	2 to 15	-	> 2
	Neighborhood	Same as for SO_2	2 to 15	-	> 2
	Background	Same as for SO_2	2 to 15	-	> 2
Ozone	Peak	Representative area downwind of the CBD[c] area 15 to 25 km from downtown and > 100 m from major traffic arteries or parking areas	3 to 15	1 to 2	> 2
	Neighborhood	Sites in center city, residential, commercial areas	3 to 15	1 to 2	> 2
	Background	Nonurban area within Region	3 to 15	1 to 2	> 2
Nitrogen dioxide	Peak	Same as for Ozone, except distance of 10 to 15 km	3 to 15	1 to 2	> 2
	Neighborhood	Same as for Ozone	3 to 15	1 to 2	> 2
	Background	Same as for Ozone	3 to 15	1 to 2	> 2
Carbon monoxide	Street Canyon	See Supplement A	3 ± 1/2	1	> 2
	Neighborhood	See Supplement A	3 ± 1/2	1	> 2
	Corridor	See Supplement A	3 ± 1/2	1	> 2
	Background	See Supplement A	3 to 10	1	> 2
Non-Methane hydrocarbons	Research and Planning	Generally limited to areas of peak emission density of hydrocarbons, i.e., CBD[c]			
Nitric Oxide	Research and Planning	Generally coincident with NMHC sites	3 to 15	1 to 2	> 2
Nitrogen Dioxide	Research and Planning	Generally coincident with NMHC sites	3 to 15	1 to 2	> 2

*Guidance for Air Quality Monitoring Network Design and Instrument Siting (Revised). U.S. Environmental Protection Agency. Guideline Series OAQPS No. 1.2-012, September 1975.[1]

[a] Vertical clearance above rooftop or other supporting structure.

[b] Horizontal clearance from side of supporting structure or other restriction to air flow.

[c] Central Business District.

1. For the most polluted sites in an urban area, sample at a frequency greater than the minimum requirements.

2. The most frequent high-volume samples should be taken at sites where the highest 24-hour and annual averages are expected.

3. If possible, use continuous monitors in place of SO_2/NO_2 impingers in areas of maximum SO_2 and NO_2 concentrations.

4. Intermittent sampling can be used at noncritical sites, i.e., sites other than maximum concentration sites.

5. For intermittent sampling, a systematic sampling schedule should be adopted considering statistical relationships for characterizing an air pollutant for a given time period and area.[6,7] Actually any schedule which gives 61 samples per year and five per quarter, in accordance with 7 below, is satisfactory but not as convenient as the systematic schedule of every 6th day, for example.

6. For downwind sites monitoring for SO_2, NO_2, and TSP from isolated point sources, use continuous instruments for the gaseous pollutants, and sample at least once every six days for TSP.

7. Minimum numbers of samples required for the appropriate summary statistics should be taken. For continuous measurements, at least 75 percent of the total possible observations must be present before summary statistics are calculated. The exact requirements follow:[7]

Time interval	Minimum number of observations
3-hour running average	3 consecutive hourly observations
8-hour running average	6 hourly observations
24-hour	18 hourly observations
Monthly	21 daily averages
Quarterly	3 consecutive monthly averages
Yearly	9 monthly averages with at least two monthly averages per quarter

For intermittent sampling data there must be at least five observations per quarter. If one month has no observations, the remaining two months must have at least two observations.

8. If validation procedures indicate that the criteria in Item 7 are not fulfilled (the minimum numbers of observations

must be valid observations), the sampling frequency should be increased during the period in which corrective measures are being pursued.

A more extensive treatment of sampling frequencies as related to data analysis is included in References 6, 7, and 8.

1.3 References

1. Guidelines of Air Quality Monitoring Network Design and Instrument Siting (Revised). U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards. OAQPS No. 1.2-012, September 1975. (Draft)
2. Air Quality Monitoring Site Description Guideline, Environmental Protection Agency, Research Triangle Park, North Carolina, OAQPS No. 1.2-019, 1974. (Draft)
3. Code of Federal Regulations 40. Protection of the Environment. Parts 50 to 69. Revised July 1, 1975.
4. Turner, D. B. Workbook of Atmospheric Dispersion Estimates. Environmental Protection Agency. 7th printing, January 1974.
5. Ludwig, F. L. and J. H. S. Kealoha. Selecting Sites for Carbon Monoxide Monitoring. EPA-450/3-75-077, September 1975.
6. Hunt, W. F. The Precision Associated with the Sampling Frequency of Log Normally Distributed Air Pollutant Measurements. JAPCA, 22. September 1972.
7. Guidelines for the Evaluation of Air Quality Data. Environmental Protection Agency, Office of Air Quality Planning and Standards. OAQPS No. 1.2-015. January 1974, P. 21.
8. Akland, G. Design of Sampling Schedule. JAPCA 22. April 1972.

2.0 SAMPLING CONSIDERATIONS

2.1 Environment Control

A proper sampling environment demands control of all physical parameters external to the samples that might affect sample stability, chemical reactions within the sampler, or sampler component function. The important parameters to be controlled are summarized in Table 2.1.

Table 2.1 ENVIRONMENT CONTROL PARAMETERS

Parameter	Source of specification	Method of control
1. Instrument vibration	Manufacturer's specifications	Design of instrument housings, benches, etc., per mfr. spec.
2. Light	Method description or manufacturer's specifications	Shield chemicals or instruments that can be affected by natural (or artificial) light.
3. Electrical voltage	Method description or manufacturer's specifications	Constant voltage transformers or regulators Separate power lines. Isolate high current drain equipment such as hi-vols, heating baths, pumps from regulated circuits.
4. Temperature	Method description or analyzer specifications	Regulated air conditioning system. 24-hour temperature recorder. Use electric heating and cooling only.
5. Humidity	Method description or analyzer specifications	Regulated air conditioning system. 24-hour recorder.

2.2 Sampling Probes and Manifolds

2.2.1 Design of Probes and Manifolds[1] - Some important variables affecting the sampling manifold design are the diameter, length, flow rate, pressure drop, and materials of construction. Considerations for these parameters are discussed below for both a vertical laminar flow and a conventional manifold design.

Vertical laminar flow design - By the proper selection of a large diameter vertical inlet probe and by maintaining a laminar flow throughout, the sample air is not permitted to react with the walls of the probe. Numerous materials such as glass, PVC plastic, galvanized steel, stainless steel, etc. can be used for this probe. Removable sample lines constructed of Teflon or glass can be used to provide each device with sample air. A flow rate of 5 liters per minute (ℓ/min) in 1.5-cm-diameter tubing (commonly used in monitoring stations) is not satisfactory for this application because of almost complete diffusion losses.

Diameters from 1.5 to 2.5 cm with 50 to 150 ℓ/min are unacceptable because of high pressure drops. Therefore, inlet line diameters of 15 cm with a flow rate of 150 ℓ/min are necessary if diffusion losses and pressure drops are to be minimized. The sampling rate should be maintained to insure laminar flow conditions. Figure 2.1 is an example of a vertical laminar flow manifold.

This configuration has the following advantages:

1. A 15-cm pipe can be cleaned easily by pulling a cloth through it with a string.
2. Sampling ports can be cut into the pipe at any location and, if unused, can be plugged with stoppers of similar compositions.
3. Metal poses no breakage hazard.
4. The pipe does not have to be clean to provide a representative sample, as in the case with smaller tubes.

Conventional manifold design - In practice it may be difficult to obtain vertical laminar flow because of necessary elbows within the intake manifold system. Therefore, a conventional manifold system employs inert materials such as Pyrex glass and/or Teflon and should be constructed in modular sections

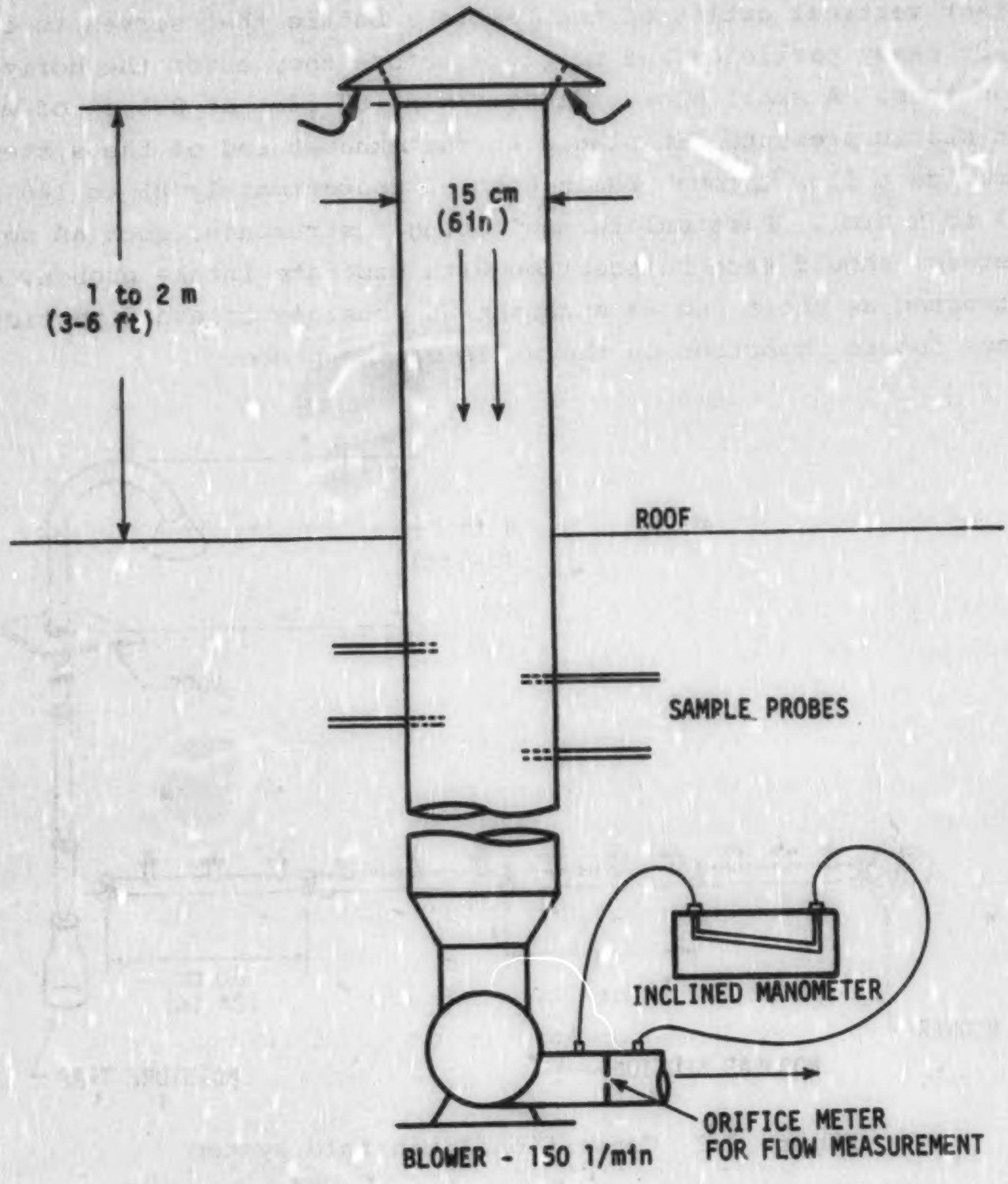

15 cm
(6in)
1 to 2 m
(3-6 ft)
ROOF
SAMPLE PROBES
INCLINED MANOMETER
ORIFICE METER
FOR FLOW MEASUREMENT
BLOWER - 150 l/min

Figure 2.1 Vertical laminar flow manifold

to enable frequent cleaning. The system (see Figure 2.2) consists of a vertical "candy cane" protruding through the roof of the shelter with a horizontal sampling manifold connected by means of a tee to the vertical section. Connected to the other vertical outlet of the tee is a bottle that serves to collect heavy particles and moisture before they enter the horizontal sections. A small blower, 1700 ℓ/min (60 cfm) at 0.0 cm of water at static pressure, is placed at the exhaust end of the system to provide a flow through the system of approximately 85 to 140 ℓ/min (3 to 5 cfm). Particulate monitoring instruments, such as nephelometers, should each be provided with separate intake probes, constructed as short and as straight as possible to avoid particulate loss due to impaction on the walls of the probe.

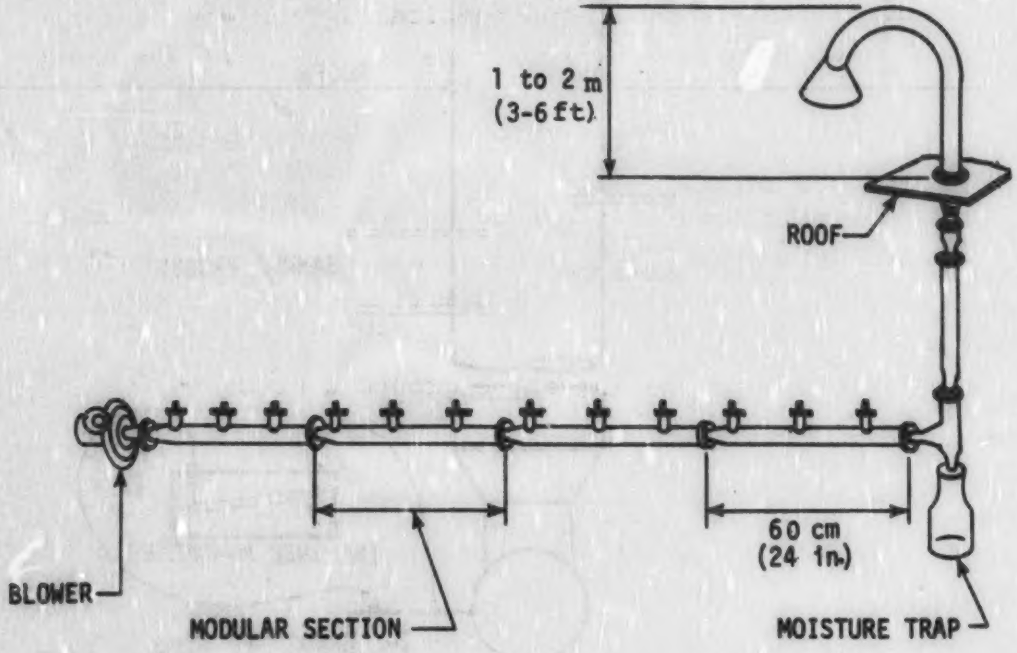


Figure 2.2 Conventional manifold system

Recent investigations[2] have shown that there are no significant losses of reactive gas (ozone) concentrations in conventional 1/2 inch inside diameter sampling lines of glass or teflon, provided that the sample residence time is 10 seconds or less. This is true even in sample lines up to 125 feet in length which collect substantial amounts of visible contamination due to ambient aerosols. However, when the sample residence time exceeds 20 seconds, loss is detectable, and at 60 seconds the loss is nearly complete. Conversely, when the sample lines are free of contamination, there is no detectable loss at sample residence times up to 120 seconds.

2.2.2 Placement of Probes and Manifolds - Probes and manifolds must be placed to avoid introducing bias to the sample. Important considerations are probe height above the ground, probe length (for horizontal probes), and physical influences near the probe. Some general guidelines for probe and manifold placement are:

1. Avoid placing probes next to air outlets such as exhaust fan openings.
2. Horizontal probes must extend beyond building overhangs.
3. Probes should not be near physical obstructions such as chimneys, etc., which can affect the air flow in the vicinity of the probe.
4. Height of the probe above the ground depends on the pollutant being measured.

2.3 Maintenance[3]

After an adequately designed sampling probe and/or manifold has been selected and installed, the following steps will help in maintaining constant sampling conditions:

1. Conduct leak test - For the conventional type manifold, seal all ports and pump down to approximately 1.25 cm (0.5 in.) water gauge vacuum as indicated by a vacuum gauge or manometer connected to one port. Isolate the system. The vacuum measurement should show no change at the end of a 15-minute period.

2. Establish cleaning techniques and schedule - a large-diameter manifold may be cleaned by pulling through it a cloth on a string. Otherwise the manifold must be disassembled periodically and cleaned with soap and water. Visible dirt should not be allowed to accumulate.

3. Plug the ports on the manifold when sampling lines are detached.

4. Maintain a flow rate in the manifold at 3 to 5 times the total sampling requirements or at a rate equal to the total sampling requirement plus 140 ℓ/min (5 cfm). This will help to reduce sample residence time in the manifold and ensure adequate gas flow to the monitoring instruments.

5. Vacuum in the manifold should not exceed 0.64 cm (0.25 in.) water gauge. Keeping the vacuum low will help to prevent the development of leaks.

2.4 Support Services

Most of the support services necessary for the successful operation of ambient air monitoring networks can be provided by the laboratory. The major support services are the generation of reagent water and preparation of standard atmospheres for calibration of equipment. Table 2.2 summarizes guidelines for quality control of these two support services.

Table 2.2 TECHNIQUES FOR QUALITY CONTROL[4]
OF SUPPORT SERVICES

Support service	Parameters affecting quality	Control techniques
Laboratory and calibration gases	Purity specifications - vary among manufacturers	Develop purchasing guides.
	Variation between lots	Overlap use of old and new cylinders
	Atmospheric interferences	Adopt filtering and drying procedures
	Composition	Traceability to primary standard
Reagents and water	Commerical source variation	Develop purchasing guides. Batch test for conductivity
	Purity requirements	Redistillation, heating, deionization with ion exchange columns
	Atmospheric interferences	Filtration of exchange air
	Generation and storage equipment	Maintenance schedules from manufacturer recommendations and from method requirements

2.5 References

1. Guidelines of Technical Services of a State Air Pollution Control Agency. Environmental Protection Agency. Contract No. 68-02-0211, 1972.
2. Unpublished Research, California Air Resource Board, January 1977.
3. Field Operations Guide for Automatic Air Monitoring Equipment. Environmental Protection Agency. APTD-0736. October 1972.
4. Quality Control Practice in Processing Air Pollution Samples. Environmental Protection Agency. APTD-1132. March 1973.

3.0 DATA HANDLING AND REPORTING

3.1 Data Recording

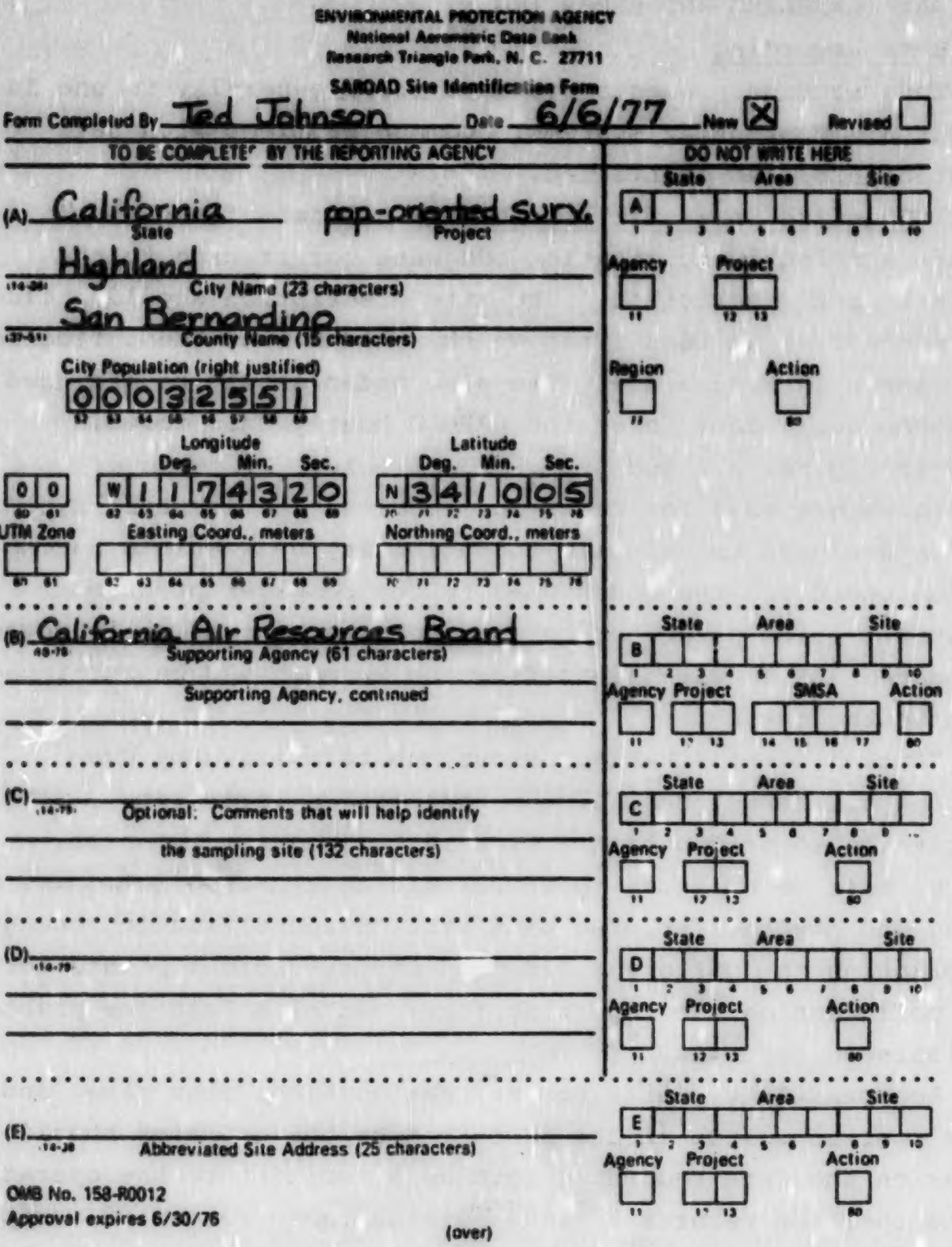
This section presents standard forms generally in use in ambient air monitoring systems. Common problems with data reporting are also identified.

3.1.1 Standard Forms for Reporting[1]- All data forms should include station identification adequate for tracing to the original site description. The site description should include all information as identified on the SAROAD Site Identification Form, shown in Figure 3.1. The same codes should be assigned to SAROAD daily data forms and SAROAD hourly data forms, shown in Figures 3.2 and 3.3, respectively. If computer techniques are used for recording results, the computer system must be designed to maintain compatibility between the SAROAD station codes and the codes used by the computer program. Whenever station parameters change, or when a station is moved, an updated SAROAD Site Identification Form should be submitted to the data bank.

Station identification errors can be avoided by pre-printing all forms, checklists, calibration forms, etc., with the station identification. If this technique is adopted, control must be employed to be certain that unused forms are discarded and new ones printed when station identification changes. Pre-printing the pollutant I.D. and proper decimal points for that pollutant on the reporting forms can eliminate the problem of misplaced decimals.

Acceptability limits for start-stop time, flow rate, and other routine system checks performed by the operator should appear on the data recording form as a reminder to the operator. If the operator records a value outside these ranges of acceptability, he should flag the value for the attention of individuals performing data validation functions.

3.1.2 Data Errors in Intermittent Sampling - The most common errors in recording data in the field are transposition of



ENVIRONMENTAL PROTECTION AGENCY
National Aerometric Data Bank
Research Triangle Park, N. C. 27711

SAROAD Site Identification Form

Form Completed By Ted Johnson Date 6/6/77 New ☒ Revised ☐

TO BE COMPLETED BY THE REPORTING AGENCY	DO NOT WRITE HERE
(A) California (State) pop-oriented surv. (Project)	A ☐☐☐☐☐☐☐☐☐ (State, Area, Site)
Highland (14-36) City Name (23 characters)	Agency ☐ Project ☐☐
San Bernardino (37-51) County Name (15 characters)	
City Population (right justified): 00032551	Region ☐ Action ☐
Longitude Deg. Min. Sec.: 00 W1174320 Latitude Deg. Min. Sec.: N341005	
UTM Zone ☐☐ Easting Coord., meters ☐☐☐☐☐☐☐☐ Northing Coord., meters ☐☐☐☐☐☐☐	
(B) California Air Resources Board (40-79) Supporting Agency (61 characters) Supporting Agency, continued	B ☐☐☐☐☐☐☐☐☐ (State, Area, Site) Agency ☐ Project ☐☐ SMSA ☐☐☐☐ Action ☐
(C) (14-79) Optional: Comments that will help identify the sampling site (132 characters)	C ☐☐☐☐☐☐☐☐☐ (State, Area, Site) Agency ☐ Project ☐☐ Action ☐
(D) (14-79)	D ☐☐☐☐☐☐☐☐☐ (State, Area, Site) Agency ☐ Project ☐☐ Action ☐
(E) (14-38) Abbreviated Site Address (25 characters)	E ☐☐☐☐☐☐☐☐☐ (State, Area, Site) Agency ☐ Project ☐☐ Action ☐

OMB No. 158-R0012
Approval expires 6/30/76

(over)

Figure 3.1 SAROAD Site Identification (front)

SAROAD Site Identification Form (continued)

TO BE COMPLETED BY THE REPORTING AGENCY

(F) 1050 San Bernardino Rd
(14 - 54) Sampling Site Address (41 characters)

Address, continued

Check the ONE major category that best describes the location of the sampling site.

Next, check the subcategory that best describes the dominating influence on the sampler within approximately a 1-mile radius of the sampling site.

1. ☐ CENTER CITY
   - ☐ 1. Industrial
   - ☐ 2. Residential
   - ☐ 3. Commercial
   - ☐ 4. Mobile

2. ☒ SUBURBAN
   - ☐ 1. Industrial
   - ☐ 2. Residential
   - ☒ 3. Commercial
   - ☐ 4. Mobile

3. ☐ RURAL
   - ☐ 1. Near urban
   - ☐ 2. Agricultural
   - ☐ 3. Commercial
   - ☐ 4. Industrial
   - ☐ 5. None of the above

4. ☐ REMOTE

Specify units 20 ft
Elevation of sampler above ground

Specify units 310 ft
Elevation of sampler above mean sea level

Circle pertinent time zone: EASTERN CENTRAL MOUNTAIN PACIFIC YUKON ALASKA BERING HAWAII

DO NOT WRITE HERE

State Area Site
F ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐
1 2 3 4 5 6 7 8 9 10

Agency ☐ 11

Project ☐ ☐ 12 13

Station Type ☐ ☐ 55 56

County Code ☐ ☐ ☐ ☐ 57 58 59 60

AQCR Number ☐ ☐ ☐ 61 62 63

AQCR Population ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ 64 65 66 67 68 69 70 71

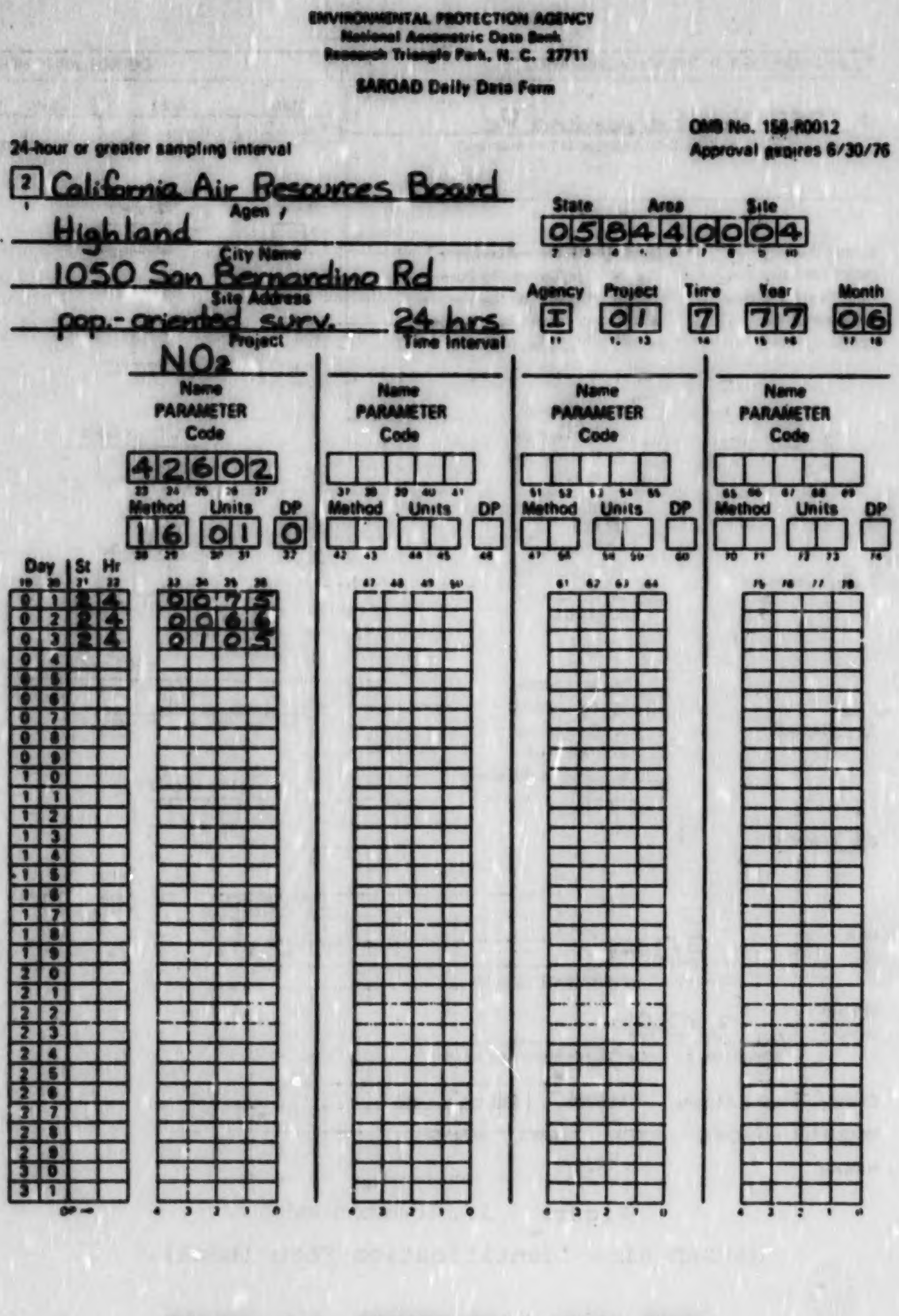
Elevation/Gr ☐ ☐ ☐ 72 73 74

Elevation/MSL ☐ ☐ ☐ ☐ 75 76 77 78

Time Zone ☐ 79

Action ☐ 80

Figure 3.1.. (continued)
SAROAD Site Identification Form (back).



ENVIRONMENTAL PROTECTION AGENCY
National Aerometric Data Bank
Research Triangle Park, N. C. 27711

SAROAD Daily Data Form

OMB No. 158-R0012
Approval expires 6/30/76

24-hour or greater sampling interval

2 California Air Resources Board
Agency

Highland
City Name

1050 San Bernardino Rd
Site Address

pop.-oriented surv.
Project

24 hrs
Time Interval

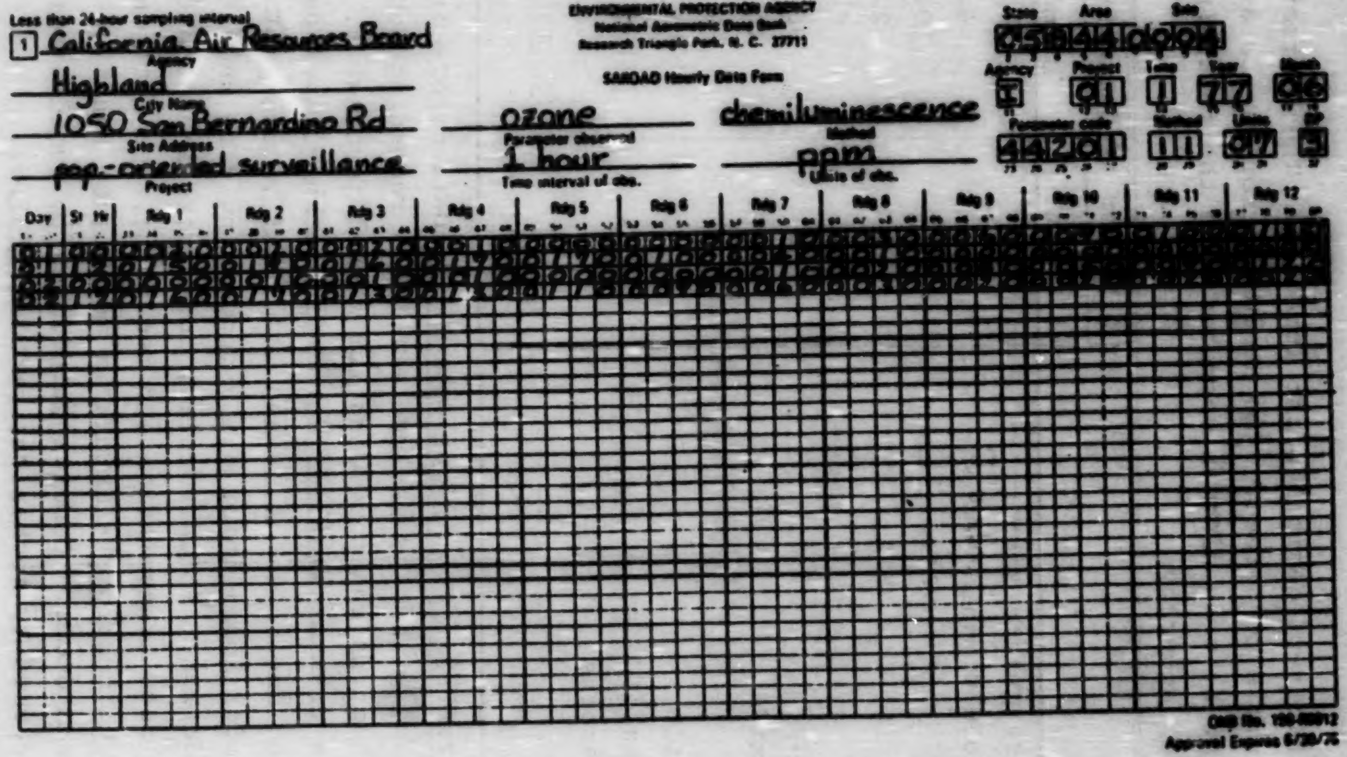
State Area Site: 0 5 8 4 4 0 0 0 4

Agency: I
Project: 0 1
Time: 7
Year: 7 7
Month: 0 6

Name	NO2				
PARAMETER Code	42602				
Method	16				
Units	01				
DP	0				

Day	St	Hr				
01	2	4	0075			
02	2	4	0066			
03	2	4	0105			
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
DP →			4 3 2 1 0	4 3 2 1 0	4 3 2 1 0	4 3 2 1 0

Figure 3.2 SAROAD Daily Data Form



Less than 24-hour sampling interval

1 California Air Resources Board
Agency

Highland
City Name

1050 San Bernardino Rd
Site Address

pop-oriented surveillance
Project

ENVIRONMENTAL PROTECTION AGENCY
National Aerometric Data Bank
Research Triangle Park, N. C. 27711

SAROAD Hourly Data Form

ozone
Parameter observed

1 hour
Time interval of obs.

chemiluminescence
Method

ppm
Units of obs.

State Area Site: 0 5 5 4 4 0 0 0 4
Agency: I
Project: 0 1
Time: 1
Year: 7 7
Month: 0 6
Parameter code: 4 4 2 0 1
Method: 1 1
Units: 0 0 7
DP: [illegible]

Day
St
Hr
Rdg 1
Rdg 2
Rdg 3
Rdg 4
Rdg 5
Rdg 6
Rdg 7
Rdg 8
Rdg 9
Rdg 10
Rdg 11
Rdg 12

OMB No. 158-R0012
Approval Expires 6/30/78

Figure 3.3 SAROAD Hourly Data Form

digits and incorrect decimal point location. These errors are almost impossible to detect. The decimal error can be avoided to some extent by providing an operator with the following guidelines for accuracy:

	Number of decimal places	
Pollutant	$\mu g/m^3$	Example
Suspended particulate matter	0	87
Benzene soluble organic matter	1	6.1
Sulfates	1	10.1
Nitrates	1	2.3
Ammonium	1	0.7
Sulfur dioxide	0	98
Nitrogen dioxide	0	40
Nitric oxide	0	8
Carbon monoxide	1	4.5
Total oxidants	0	100
Total hydrocarbons	1	2.7
Ozone	0	72
Methane	1	1.5

3.1.3 Data Errors in Continuous Sampling - Data errors in continuous sampling primarily include errors in recording device function or in strip chart reading for manual techniques, and errors in data transmission for automatic techniques of data recording. See Sections 1.4.10 and 1.4.17 of Volume I of this Handbook for additional information concerning data reduction and data validation. In particular, for errors in automatic techniques of data transmission and recording, see Section 1.4.10.

Strip chart errors - Errors in mechanical function of strip charts can occur. General guidelines to avoid errors or loss of data caused by mechanical problems follow:

1. Perform a daily check to assure an adequate supply of strip chart paper. Check the ink level in the recorder pen to verify that the level is adequate for the next sampling period and that the pen tip is not blocked.

2. Perform a daily check to verify that the pen on the recorder aligns with the baseline of the strip chart during the instrument zero check.

3. Verify the timing of the strip chart drive against a standard time piece. This should be accomplished immediately after installation of the recorder, and at intervals dictated by experience with the recorder.

4. Replace recorder pens and soak in cleaning solution occasionally.

5. Examine the strip chart for apparent evidence of chart drag or malfunction and mark suspected intervals.

When reviewing a strip chart, typical signs of system malfunction are:

1. A straight trace for several hours (other than minimum detectable).

2. Excessive noise as indicated by a wide solid trace, or erratic behavior such as spikes that are sharper than is possible with the normal instrument response time. Noisy outputs usually result when analyzers are exposed to vibration sources.

3. A long steady increase or decrease in deflection.

4. A cyclic pattern of the trace with a definite time period indicating a sensitivity to changes in temperature or parameters other than the pollutant concentration.

5. Periods where the trace drops below the zero baseline. This may result from a larger-than-normal drop in the ambient room temperature or power line voltage.

Void any data for any time interval for which malfunction of the sampling system is detected.

Suggestions for minimizing errors in reading strip charts are as follows:

1. Chart readers should be trained with a standard strip of chart, whose readings have been determined by one or more experienced readers. When the new reader can perform adequately on the standard strip then permit him to read new sample charts. A suggested procedure for reading a strip chart is given in the following subsection.

2. An individual should spend only a portion of a day reading strip charts. A drop in productivity and reliability is expected after a few hours.

3. A senior technician should check at least 5 to 10 percent of the strip chart values reduced. The percent of checking is arbitrary and depends on the depth of training of the junior technicians and the time available for checking their work. If minimum performance criteria established for a particular network are not being met, additional training is indicated.

4. Use a chart reader to reduce technician fatigue and improve accuracy in data reduction.

Data reduction from strip chart - To obtain hourly average concentrations from a strip chart record, the following procedures are used:

1. The strip chart record for the sampling period must have span and zero traces at the beginning and at the end of the sampling period.

2. Fill in the identification data called for at the top of an Hourly Averages Sheet as illustrated in Figure 3.4.

3. Using a straight edge, draw a line from the zero baseline at the start of the sampling period to the zero baseline at the end of the sampling period. This line represents the zero baseline to be used for the sampling period.

4. Read the zero baseline in percent of chart at the midpoint of each hour interval and record the value on Figure 3.4.

5. Determine the hourly averages by using a transparent straight edge at least 1 in. long. Place the straight edge parallel to the horizontal chart division lines. For the interval of interest between two vertical hour lines, adjust the straight edge between the lowest and highest points of the trace in that interval, keeping the straight edge parallel to the chart division lines until the total area above the straight edge bounded by the trace and the hour lines is estimated to equal the total area below the straight edge bounded by the trace and hour lines.

CITY Highland, California
SITE LOCATION 1050 San Bernardino Rd
CHECKER Beth Feagin

SITE NO. 7
POLLUTANT ozone (ppm)
OPERATOR Ted Johnson

		READING		ZERO BASELINE		DIFFERENCE		ADD + 5		ppm	
DATE	HOUR	ORIGINAL	CHECK	ORIGINAL	CHECK	ORIGINAL	CHECK	ORIGINAL	CHECK	ORIGINAL	CHECK[a]
6/15	00	9		5		4		9		0.020	

[a] The check columns are for use by an independent audit. The check is performed on approximately 7 percent of the data.

Figure 3.4 Sample sheet for recording hourly averages

Read and record the percentage of chart deflection on the Hourly Average Sheet. Repeat the procedure for all the hour intervals sampled and which have not been marked invalid. Record all values on Figure 3.4 in the column headed "Reading - Original".

6. Subtract the zero baseline value, from the reading value and record the difference on Figure 3.4.

7. Add the percent zero offset, +5, to the difference on Figure 3.4.

8. Convert percentage chart values to concentration in ppm using the most recent calibration curve. Record the ppm NO_2 values in the last column of Figure 3.4.

Data validation - The purpose of data validation is to detect and then correct any data values that may not represent actual air quality conditions at the sampling station. Effective data validation procedures usually are handled completely independently from the procedures of initial data collection. Moreover, it is advisable that the individuals responsible for data validation not be directly involved with data collection.

Both manual and computer-oriented systems require individual review of all data tabulations. As an individual scans tabulations, there is no way that he can determine that all values are, or are not, valid. The purpose of manual inspection is to spot unusually high (or low) values that might indicate a gross error in the data collection system. Obviously, to recognize that the reported concentration of a given pollutant is extreme, the individual must have basic knowledge of the major pollutants and of air quality conditions prevalent at the reporting station. Data values considered questionable are referred to the data collection group for verification. This scanning for high/low values is sensitive to spurious extreme values but not to intermediate values that could be grossly in error.

Manual review of data tabulations also allows detection of uncorrected drift in the zero-baseline of a continuous sensor. Zero drift may be indicated when the daily minimum concentration tends to increase or decrease from the norm over a period of several days. For example, at most sampling stations the early morning (3:00 to 4:00 A.M.) concentrations of carbon monoxide tend to reach a minimum of 2 to 4 ppm. If the minimum concentration differs significantly from this, a zero-drift can be suspected. Zero-drift could be confirmed by review of the original strip chart.

In an automated data processing system, procedures for data validation can easily be incorporated into the basic software. The computer can be programmed to scan each data value before preparing an output tabulation. Questionable data values are then flagged on the data tabulation to indicate a possible error.

A computer can easily handle inspection for extreme values. Tables of high and low values for each pollutant at each sampling station can be built into the program. The check for extreme values can be further refined to account for time of day, time of week, and other cyclic conditions.

Because the computer can perform computations and make comparisons extremely rapidly, it can also make some determination concerning the validity of data values that are not necessarily high or low. Data validation procedures should be recommended as standard operating procedure.[2] One way to do this is to test the difference between successive data values, since one would not normally expect very rapid changes in concentrations of a pollutant during a 5-minute or 1-hour reporting period. When the difference between two successive values exceeds a predetermined value, the tabulation can be flagged, with an appropriate symbol. For example, if two values for hourly sulfur dioxide concentrations differ by more than 0.05 ppm (100 $\mu g/m^3$), the data validation staff may wish to recheck the original records from which the data were obtained.

3.3 Systematic Data Management

An orderly process of data management based on analysis of all the data-handling procedures and their interrelationships is sometimes called a "systems approach". This kind of systematic overview of the total data function is accomplished in three phases:

1. Surveying current and future reporting requirements.
2. Outlining the present routine flow of data within and outside the agency.
3. Redesigning the current system to allow maximum functional overlap of filing and retrieval routines.

A survey of current reporting requirements involves summarizing and categorizing the reports currently required and their important data elements. The purpose of this analysis is to identify report elements that require similar input, to allow optimum scheduling, and to differentiate between required reports and those provided as a service. Future reporting requirements will be based on projected legal requirements, projected development of systems to communicate with various data banks, and projected growth of the air quality surveillance network.

Outlining present data flow requires a review of the origin of each data form, the editing procedures applied, the calculations performed, the application of quality control procedures, and the reports for which each form is used. The purpose of outlining the data flow is to identify data elements that are subjected to similar checking and calculating procedures and to classify them according to their point of origin. Once again, this procedure provides a means of preventing unnecessary duplication.

As a final step in systematic data management, the data system should be continually updated. The following items are suggested for review:

1. What operations are duplicated in the system?
2. How can the system be changed to eliminate needless duplications?
3. How do the manual systems and computerized systems augment each other?
4. Are the data formats, identification codes, and other elements compatible throughout the system?
5. Can reporting schedules be changed to minimize filing and retrieval of each data record?
6. Can special techniques, such as the use of multipart forms, be applied to minimize data transposition?

7. Are filing and retrieval systems sufficiently flexible to allow expansion or upgrading at minimum cost?

3.4 Evaluation of Air Quality Data

Minimum requirements and procedures for evaluation of air quality data are given in references 2, 3, and 4. A basic discussion of the statistical methods is contained in the Appendix to Volume I of this Handbook[5]. However, the analyst should also use reference 4 for specific statistical methodology for analysis of air quality trends.

3.5 References

1. Quality Control Practice in Processing Air Pollution Samples. Environmental Protection Agency. APTD-1132. March 1973.
2. Procedures for Flow and Auditing of Air Quality Data. Environmental Protection Agency, Research Triangle Park, N. C. OAQPS No. 1.2-013. May 1974.
3. Guidelines for the Evaluation of Air Quality Trends. Environmental Protection Agency, Research Triangle Park, N. C. OAQPS No. 12-014. Dec. 1974.
4. Guidelines for the Evaluation of Air Quality Data. Environmental Protection Agency, Research Triangle Park, N. C. OAQPS No. 1.2-015. Jan. 1975.
5. Quality Assurance Handbook for Air Pollution Measurement Systems. Volume I - Principles, EPA-600/9-76-005, March 1976.

4.0 COMPLIANCE MONITORING - REFERENCE AND EQUIVALENT METHODS

Certain air pollution control agencies, State agencies, for example, are required to monitor the ambient air to determine compliance with the National Ambient Air Quality Standards (NAAQS). These standards are given in Title 40, Chapter 1, Part 50 of the Code of Federal Regulations (40 CFR Part 50).[1] The specific monitoring requirements are provided in 40 CFR Part 51, "Requirements for Preparation, Adoption, and Submittal of Implementation Plans". Section 51.17a, an amendment to Part 51, promulgated on February 18, 1975 (40 FR 7043), requires that for purposes of section 51.17 (i.e., compliance monitoring) "...each method for measuring SO_2, CO, or photochemical oxidant... shall be a reference method or equivalent method...." An exception* to this requirement allows analyzers (automated methods) purchased prior to February 18, 1976, to be used until February 18, 1980. Also, as of this writing, this requirement is in the process of being extended to apply to methods for measuring ambient NO_2.

The definitions and qualifications of reference and equivalent methods are given in 40 CFR Part 53. However, for compliance monitoring, the distinction between reference and equivalent methods is relatively unimportant, either may be used interchangeably.

Reference and equivalent methods may be either manual or automated methods (analyzers). For SO_2, the reference method is a manual method and is completely specified in Appendix A of 40 CFR Part 50. Thus, all other methods for SO_2 qualify for designation only as equivalent methods. For CO, NO_2, and oxidants, Part 50 gives only a measurement principle and calibration procedure applicable to reference methods for those pollutants. Automated methods (analyzers) for those pollutants may be designated either as reference methods or equivalent methods, depending on whether or not the method utilizes the measurement principle and calibration procedure specified for reference methods. Since any analyzer which meets the requirements of the specified measurement principle and calibration procedure may be designated as a reference method, there may be numerous reference methods for CO, NO_2, or oxidants. Further information on this subject may be obtained from the preamble to 40 CFR Part 53, published February 18, 1975 (40 FR 7045-7046).

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*There are other exceptions; see Amendment to Part 51 promulgated March 17, 1976 (Federal Register, Vol. 41, pages 11253-5) for specific information.

Except for the (unique) reference method for SO_2 noted above, all reference and equivalent methods must be officially designated as such by EPA. Notice of each method designated is published in the Federal Register at the time of designation. In addition, a current list of all designated reference and equivalent methods is maintained by EPA and updated whenever a new method is designated. This list may be obtained from any EPA Regional Office or from the Environmental Monitoring and Support Laboratory, Department E, MD-76, Research Triangle Park, North Carolina 27711. Moreover, any analyzer offered for sale as a reference or equivalent method after April 16, 1976, must bear a label or sticker indicating that the analyzer has been designated as a reference or equivalent by EPA.

For automated methods, a designation applies to any analyzer which is identical to the analyzer described in the designation. In many cases, analyzers manufactured prior to the designation may be upgraded (e.g., by minor modification or by substitution of a new operation or instruction manual) so as to be identical to the designated method and thus achieve designated status at modest cost. The manufacturer should be consulted to determine the feasibility of such upgrading. Any modification to a reference or equivalent method made by a user must be approved by EPA if the designated status is to be maintained (see 40 CFR

51.17a(f), promulgated on March 17, 1976 in the Federal Register, Vol. 41, page 11255).

Sellers of designated methods must comply with several conditions summarized below:

1. A copy of the approved operation or instruction manual must accompany the analyzer when it is delivered to the ultimate purchaser.

2. The analyzer must not generate any unreasonable hazard to operators or to the environment.

3. The analyzer must function within the limits of the performance specifications given in Table 4.1 for at least one year after delivery when maintained and operated in accordance with the operation manual.

4. Any analyzer offered for sale as a reference or equivalent method must bear a label or sticker indicating that it has been designated as a reference or equivalent method in accordance with Part 53.

5. If such an analyzer has one or more selectable ranges, the label or sticker must be placed in close proximity to the range selector and indicate which range or ranges have been designated as reference or equivalent methods.

6. An applicant who offers analyzers for sale as reference or equivalent methods is required to maintain a list of ultimate purchasers of such analyzers and to notify them within 30 days if a reference or equivalent method designation applicable to the analyzers has been cancelled or if adjustment of the analyzers is necessary under 40 CFR 53.11(b) to avoid a cancellation.

7. An applicant who modifies an analyzer previously designated as a reference or equivalent method is not permitted to sell the analyzer (as modified) as a reference or equivalent method (although he may choose to sell it without such representations), nor to attach a label or sticker to the analyzer

Table 4.1 PERFORMANCE SPECIFICATIONS FOR AUTOMATED METHODS*

	Performance Parameter	Units[1]	Sulfur Dioxide	Oxidants	Carbon Monoxide	Definitions and Test Procedures
1.	Range	Parts per million	0-0.5	0-0.5	0-50	§53.23(a).
2.	Noise	do	0.005	0.005	0.50	§53.23(b).
3.	Lower detectable limit	do	0.01	0.01	1.0	§53.23(c).
4.	Interference Equivalent					§53.23(d).
	Each interferent	do	±0.02	±0.02	±1.0	
	Total interferent	do	0.06	0.06	1.5	
5.	Zero drift, 12 & 24 hr	do	±0.02	±0.02	±1.0	§53.23(e).
6.	Span drift, 24 hours					
	20% of upper range limit	Percent	± 20.0	± 20.0	±10.0	§53.23(e).
	80% of upper range limit	do	± 5.0	± 5.0	± 2.5	§53.23(e).
7.	Lag time	Minutes	20	20	10	§53.23(e).
8.	Rise time	do	15	15	5	§53.23(e).
9.	Fall time	do	15	15	5	§53.23(e).
10.	Precision					§53.23(e).
	20% of upper range limit	Parts per million	0.01	0.01	0.5	
	80% of upper range limit	do	0.015	0.01	0.5	

[1] To convert from parts per million to micrograms per cubic meter at 25°C and 760 mm Hg, multiply by M/0.02447, where M is the molecular weight of the gas.

*Table 4.1 is reproduced from Table B-1 of CFR 40 Part 53.

(as modified) under the provisions described above, until he has received notice under 40 CFR 53.14(c) that the original designation or a new designation applies to the method as modified or until he has applied for and received notice of a new reference or equivalent method determination for the analyzer as modified.

Aside from occasional breakdowns or malfunctions, consistent or repeated non-compliance with any of these conditions should be reported to EPA at the address given previously.

In selecting the designated methods, keep in mind that designation of a method indicates only that it meets certain minimum standards. Competitive differences still exist among analyzers. Some analyzers or methods may have performance, operational, economic or other advantages over other analyzers or methods. Thus the need for a careful selection process based on the individual air monitoring situation is still very important. However, the performance tests and other criteria used to qualify a method for designation as a reference or equivalent method are designed and intended only as GO/NO GO tests to determine compliance with the minimum standards. Therefore, this test data should not be used in a quantitative manner to compare one method with another method. Furthermore, designation as a reference or equivalent method provides no guarantee that a particular analyzer will operate properly, as any analyzer can malfunction. So an on-going quality control program is as necessary for designated methods as for any other methods.

Many organizations have elected to specify or select designated reference or equivalent methods even for non-compliance monitoring. This practice may offer significant advantages, such as (1) ease of specification, (2) guarantee of minimum performance, (3) better instruction manuals, (4) flexibility of application, and (5) increased credibility of measurements.

4.1 References

1. Code of Federal Regulations 40. Protection of the Environment. Parts 50 to 69. Revised July 1, 1975.

5.0 RECOMMENDED QUALITY ASSURANCE PROGRAM FOR AMBIENT AIR MEASUREMENTS

In order for air monitoring data to be useful, they must be of acceptable quality. The dissemination and use of data of poor or unknown quality can lead to incorrect decisions with regard to environmental standards and regulatory actions. The gathering of air monitoring data under the umbrella of a quality assurance program does much to avoid regulatory mistakes, and all control agencies should vigorously pursue the implementation of such measures.

The major elements of a satisfactory quality assurance program are (1) the availability of an evaluated measurement methodology which is adequate for its intended purpose, (2) satisfactory performance by organizations collecting the air pollution monitoring data, (3) documentation of quality assurance practices, and (4) the availability of competent technical assistance for organizations needing to improve their performance. It is imperative that the management of the monitoring program be committed to a quality assurance program and that adequate resources be available to carry on the activities involved in its major elements.

To be sure, the implementation of a formal quality assurance program does have its price, but the cost of collecting good air monitoring data is far less than the cost of making incorrect regulatory decisions because of poor data. Experience has shown that an agency should be prepared to spend between 10 and 25 percent of its monitoring budget for its quality assurance programs.

5.1 Development and Implementation of Air Pollution Measurement Quality Assurance Programs

Federal, state, and local agencies all have important roles to play in developing and implementing satisfactory quality assurance programs. EPA's responsibility is to develop the tools needed to carry on a quality assurance program, and it is up to the state and local agencies to implement the programs.

5.1.1 Role of EPA Headquarters - EPA headquarters has the following responsibilities:

1. To be certain that the methods and procedures used in making air pollution measurements are well evaluated and that their limits of precision and accuracy are well understood.
2. To determine the performance of laboratories making air pollution measurements of importance to the regulatory process.
3. To implement satisfactory quality assurance programs over EPA's air pollution monitoring which have the potential for generating data used for setting standards.
4. To be certain that air monitoring data of importance to the regulatory process is of satisfactory quality.
5. To share with EPA regional offices the rendering of technical assistance to the air pollution monitoring community.

5.1.2 Role of the EPA Regional Offices - The major responsibility of EPA's regional offices is the coordination of quality assurance matters between the various elements of EPA and the state and local agencies. This role requires that the regional offices make available to the state and local agencies the technical information and quality assurance programs which EPA headquarters has developed and make known to EPA headquarters the unmet quality assurance needs of the state and local gencies. Another very important function of the regional office is the evaluation of the ability of state and local agency laboratories to measure air pollutants of regulatory concern. To be effective in these roles, the regional offices should maintain and strengthen their technical capabilities with respect to air pollution monitoring.

5.1.3 Role of State and Local Agencies - The major responsibility of state and local agencies is the implementation of satisfactory quality assurance programs over the monitoring which yields the air quality data needed for the regulatory process. It is the responsibility of state and local agencies to implement these programs in their own laboratories and in any consulting and contractor laboratories which they may use to obtain data of importance to the regulatory process.

5.2 Minimum Quality Assurance Programs

Comprehensive quality assurance programs in air monitoring are relatively new, and many agencies responsible for air monitoring have not formalized their quality assurance activities into an identifiable program. As an aid to agencies who are developing quality assurance programs, and to agencies who wish to review existing programs, we present here those activities considered to be essential in an air pollution monitoring quality assurance program. These essential activities and other aspects of a complete quality assurance program are described in detail in "Quality Assurance Handbook for Air Pollution Measurement Systems - Volume I, Principles (EPA-600/9-76-005), and this document should be consulted in establishing or evaluating a quality assurance program.

A suggested sequence for the development of a quality assurance program is given below. About 12 months is required for complete implementation.

5.2.1 Develop Immediately -

1. Agency Quality Assurance Policy and Objective - Each agency should develop a written quality assurance policy, and this policy should be made known to all agency personnel. As a minimum, this policy should create an

awareness of quality assurance activities, provide specific procedures for implementing a quality control program, provide for corrective action when required, state quality assurance objectives for each major monitoring project operated by the agency, and explicitly delegate authority to implement quality assurance systems planned by management officials.

2. Organization and Responsibilities - An organization chart showing the key agency personnel and their area of quality assurance responsibility should be prepared. A Quality Assurance Coordinator should be designated for the agency. This designee should be responsible for the coordination of quality assurance activities within the agency and with other agencies.

5.2.2 Develop Within Six Months -

1. Measurement Method Review and Application - All existing methods (sampling and analysis) used for routine measurements should be reviewed and revised if necessary; written procedures should be prepared where none exists. A document control system should be developed for these methods to keep agency personnel abreast of changes in methodology. Any ambient air monitoring for criteria pollutants conducted under State Implementation Plans must use EPA's reference methods or EPA-approved equivalent methods.

2. Calibration Procedure Review - Calibration procedures used for all measurement methods should be reviewed, revised if necessary, documented, and included in the method write-up just mentioned. Document control should also be established for these calibration procedures to inform agency personnel of any changes. As an agency policy, traceability of the accuracy of working calibration standards should be established by comparing these standards to standards of higher accuracy whenever standards of higher accuracy are available.

3. Internal Quality Control Procedures - The procedures used during sampling and analysis to detect, correct, and

record out-of-control conditions should be defined and documented. Use of control charts is encouraged.

4. Audit Procedure - Procedures should be selected and implemented that will permit comparison of the performance of the measurement system (sampling and analysis) under routine operation versus an independent technique. Commonly, this independent technique is either a performance audit or the use of a dual measurement system. Results from these audit procedures are useful in detecting bias in the routine measurement system.

5. Interlaboratory Testing - Each agency and its contractors conducting monitoring activities should participate in the EPA quality assurance performance surveys. Requests for participation should be made at the EPA regional office.

5.2.3 Develop Within Twelve Months -

1. Data Validation Procedures - The criteria used to validate air monitoring data should be documented and the routine tests or checks on the raw data should be defined.

2. Preventive Maintenance - By measurement method, a schedule for preventive maintenance should be prepared that identifies the maintenance tasks and frequency required. A procedure for performing the maintenance task should be prepared if none is available.

3. Review of Training Needs - Proper training of staff members is essential for the performance of their assigned job responsibility. During the first twelve months, the training and experience of all staff members should be reviewed and plans made to obtain needed training.

SECTION 2.1

REFERENCE METHOD FOR THE DETERMINATION OF SULFUR DIOXIDE IN THE ATMOSPHERE (PARAROSANILINE METHOD)

OUTLINE

	Section	Documentation	Number of Pages
SUMMARY			
METHOD DESCRIPTION			
1.	PROCUREMENT OF EQUIPMENT AND SUPPLIES	2.1.1	3
2.	CALIBRATION OF EQUIPMENT	2.1.2	37
3.	PREPARATION OF REAGENTS	2.1.3	8
4.	SAMPLING PROCEDURE	2.1.4	18
5.	ANALYSIS OF SAMPLES	2.1.5	9
6.	DATA REDUCTION, VALIDATION AND REPORTING	2.1.6	4
7.	MAINTENANCE	2.1.7	2
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10.	REFERENCE METHOD	2.1.10	7
11.	REFERENCES	2.1.11	2
12.	DATA FORMS	2.1.12	14

SUMMARY

Sulfur dioxide is absorbed from ambient air into a solution of potassium tetrachloromercurate (TCM). A dichlorosulfitomercurate complex, which resists oxidation by oxygen in the air, is formed. This complex is stable to strong oxidants, such as ozone and nitrogen oxides, but is thermally unstable above 25°C. As a result of this thermal instability, the sampling and subsequent sample handling should be performed at room temperature or below. Thermally controlled sampling equipment is commercially available for this purpose. The dichlorosulfitomercurate complex is reacted with pararosaniline and formaldehyde to form an intensely colored pararosaniline methyl sulfonic acid dye. The absorbance of this solution is measured spectrophotometrically. The method can be used to determine sulfur dioxide levels in the ambient air, employing various sampling periods ranging from 30 minutes to 24 hours. The detection limit for the method as presented is 25 μg SO_2 per cubic meter or 0.01 part per million (95 percent confidence level). This detection limit can be reduced to 5 $\mu g/m^3$ by employing larger aliquots for analyses or by the use of autoanalyzer techniques.[2]

Based on more extensive review and on the experience gained in collaborative tests[2,3,4] and in audits, the method description and the limits recommended in quality checks and audits herein are in some cases different from and supercede those given in the guideline document.[1] On the other hand, Reference 1 contains functional and error analyses which are not included in this Handbook.

Blank data forms are provided in Section 12 for the convenience of the Handbook user. The reference method as given in the Code of Federal Regulations 40, Protection of the Environment, Part 50.11, July 1, 1975, is contained in Section 10.

Recommended equivalent methods are published in the Federal Register and quarterly in Analytical Quality Control Newsletter, U.S. Environmental Protection Agency, Monitoring and Support Laboratory, Cincinnati, Ohio, 45268. See Section 2.0.4 of this volume of the Handbook for a discussion of equivalency concepts.

METHOD DESCRIPTION

1.0 PROCUREMENT OF EQUIPMENT AND SUPPLIES

Specifications for the sampling equipment, analytical and support supplies for monitoring SO_2 in ambient air are presented in Section 2.1.10, the Reference Method. A tabulation of these materials is presented below with the reference to subsections of this section of the Handbook as a guide to the user for determining the specific information required. The quality assurance functions are summarized in Table 1.1.

Item	Reference to subsections within this section
Calibration Information	
Wet test meter	2.1.1
Soap bubble meter	2.1.2, 2.2.2
Mass flow meter	2.1.3, 2.2.1
Rotameter	2.1.4, 2.2.3
Critical flow orifice	2.2, 2.2.1-2.2.3
Analytical balance	2.3
Elapsed time meter	2.4
Timer	2.5
Preparation of Reagents	3.1-3.16
Sampling Procedure	
Absorber	4.2.1-4.2.4, 4.2.6-4.2.8
24-hour bubbler	4.2.7
Critical orifice	4.2.8
Vacuum pump	4.3.1, 4.3.2
Timer	4.3.1, 4.3.2
Analysis of Samples	
Absorber	5.1
Critical orifice	5.2.1
Rotameters	5.2.2
Spectrophotometer	5.3, 5.4
Maintenance	
24-hour bubbler train	7.1
Short-term sampling train	7.2

Upon receipt of the sampling equipment, apparatus, and supplies procurement checks, as described in this document, should be performed. The checks are summarized in Table 1.1, Activity Matrix for Procurement of Equipment and Supplies. All pertinent procurement, disposition and check information should be recorded in a log, an example of which is presented in Figure 1.1. This log will serve as a permanent record for future procurement needs, provide continuity of equipment and supplies; and provide a basis for future program fiscal projection.

Table 1.1 ACTIVITY MATRIX FOR PROCUREMENT OF EQUIPMENT AND SUPPLIES

Equipment	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Absorber	As per Reference Method, Section 10, Subsection 5.1.1	Upon receipt	Return to supplier
Pump	Capable of maintaining an air pressure differential greater than 0.7 atmospheres at the desired rate	Upon receipt check performance	Return to supplier
Air flow meter or critical orifice	Capable of measuring air flow within ±2%. See Section 10 for specifications for different sampling periods	Calibration checks updn receipt. See Sections 2.1.4 and 2.1.5	Adjust as per manufacturer's instructions or return to supplier
Spectrophotometer	Suitable for measurement of absorbance at 548 nm with an effective spectral band width of less than 15 nm	Upon receipt verify wavelength calibration of instrument	Request calibration check by manufacturer/supplier
Wet test meter Soap bubble meter Mass flow meter Rotameter Analytical balance Elapsed time meter Timers	See Table 2.4, at the end of Section 2, for acceptance limits relative to calibration checks	Upon receipt conduct calibration check	See Table 2.4 for action

Item description	Qty	Purchase order No.	Vendor	Date		Cost	Disposition	Comments
				Ord	Rec			
Vacuum pump Model 107 CA 20	4	01579	Thomas Industries	3/27/75	5/13/75	$52	O.K.	

Figure 1.1 Example of a procurement log

2.0 CALIBRATION OF EQUIPMENT

Prior to the implementation of a sampling and analysis program for SO_2, a wide spectrum of sampling and analysis equipment must be calibrated. The calibration activities are summarized in Table 2.4, Activity Matrix for Calibration of Equipment, located at the end of this section. Many of these calibration checks should also be employed for the initial procurement check activities. All data and calculations involved in these calibration activities should be recorded in a separate calibration log book. It is suggested that this log be arranged such that a separate section is designated for each apparatus and sampler employed in the program.

2.1 Calibration of Flow Measurement Equipment

The analytical method for the determination of sulfur dioxide in ambient air employs two basic sampling trains. The basic differences between the two sampling trains are the flow rate, impinger design, and flow control system. The sampling train for short periods, one to three hours, requires the use of a midget impinger train. The 24-hour sampler employs an impinger train of a different design. Typically these sampling trains require flow measurement equipment consisting of wet test meters, soap bubble meters, mass flow meters, and rotameters, all of which require calibration.

2.1.1 Calibration of Wet Test Meters at STP - Wet test meters are calibrated by the manufacturer to an accuracy of ± 0.5%. The calibration of the wet test meter must be checked initially upon receipt and quarterly thereafter.

The following liquid positive displacement technique can be used to verify and adjust, if necessary, the accuracy of the wet test meter to ± 1%.

1. Level the wet test meter by adjusting the legs until the bubble is centered on the level located on the top of the meter.

2. Adjust the water volume in the meter such that the pointer in the water level gauge just touches the meniscus.

3. Adjust the water manometer to zero by moving the scale or by adding water to the manometer.

4. Set up the apparatus and calibration system as shown in Figure 2.1.
   a. Fill the 5-gallon jug with distilled water to below the air inlet tube and allow to equilibrate to room temperature (about 24 hours) before use.
   b. Start water siphoning through the system at a flow rate of approximately 200 ml/min and collect the water in a one gallon container, located in place of the volumetric flask.

5. Check the physical operation of the meter as follows:
   a. If the manometer reading is less than 10 mm H_2O the meter is in proper working condition. Continue to step 6.
   b. If the manometer reading is greater than 10 mm H_2O the wet test meter is defective. Return the meter to the manufacturer for repair if the defect(s), for example, bad connections or joints, cannot be found and corrected.

6. Continue the operation until the one gallon container is almost full. Turn the siphon system off with the pinch clamp.

7. Read the initial volume (V_i) from the wet test meter dial and record on the wet test calibration log, Figure 2.2.

8. Place a clean, dry 2000 ml volumetric flask (Class A) under the siphon tube, open the pinch clamp and fill volumetric flask to the 2000 ml mark.

9. While the water is flowing, record the wet test manometer reading. Record the following additional data:
   a. Meter fluid temperature, °C.
   b. Barometric pressure, mm Hg.
   c. Volume as indicated on the meter, liters.

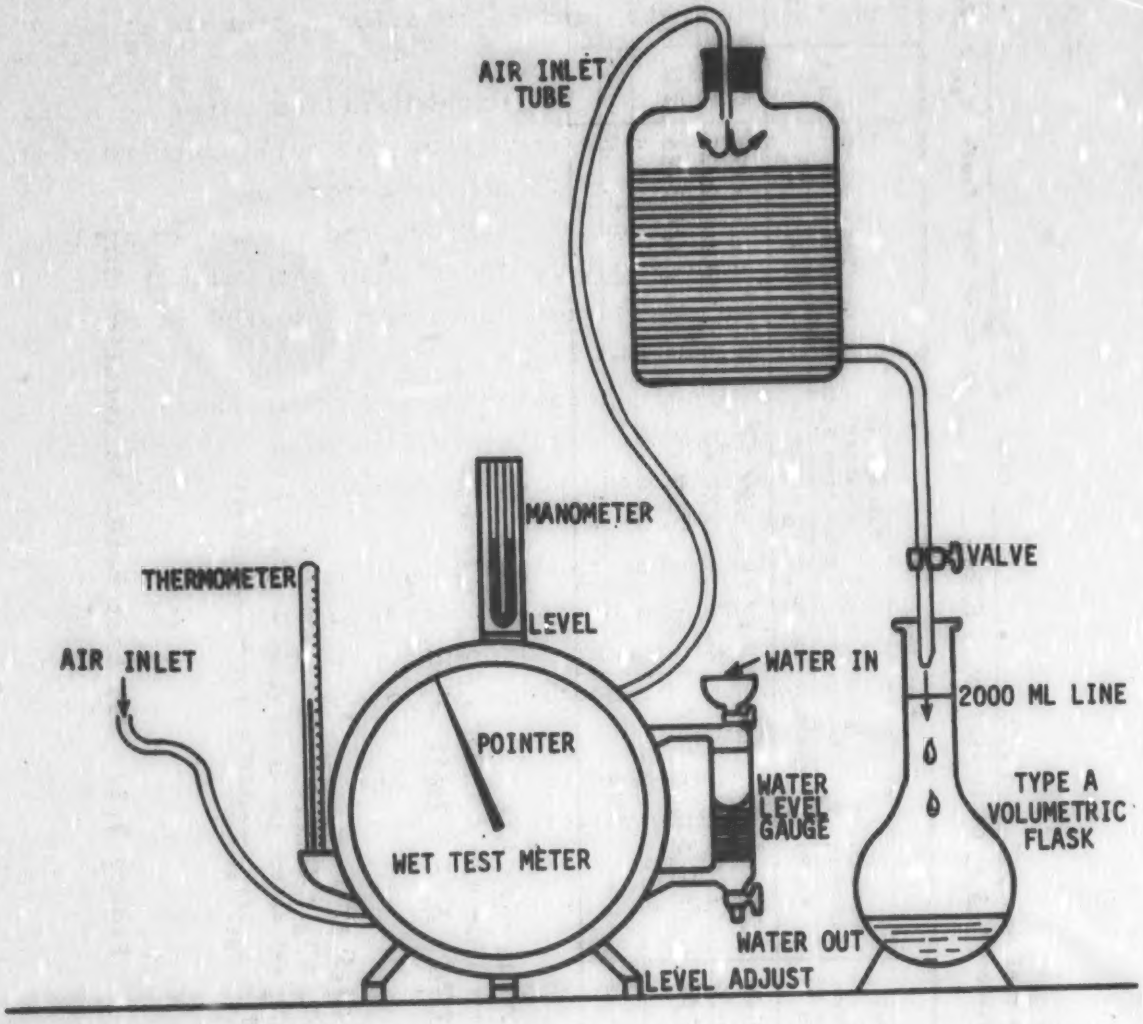
AIR INLET TUBE
MANOMETER
VALVE
THERMOMETER
LEVEL
AIR INLET
WATER IN
2000 ML LINE
POINTER
WATER LEVEL GAUGE
TYPE A VOLUMETRIC FLASK
WET TEST METER
WATER OUT
LEVEL ADJUST

Figure 2.1 Calibration check apparatus for wet test meter

Wet Test Meter Serial No. 12-310 Date 6/13/76

Wet Test Meter Range 0-3ℓ Calibrated by E.B. Volume of Test Flask, V_{st} = 2.0ℓ

Test No.	Wet Test Meter Temperature		Test Data: Barometric Pressure, P_B	Test Data: Manometer Reading, Δp		Test Data: Barometric Pressure Corrected P_B-Δp	Wet Test Meter Readings, ℓ			V_s* ℓ	Remarks
	°C	T_m in °K	mm Hg	mm H_2O	mm Hg	P_m in mm Hg	V_f	V_i	V_m		
1	22.5	295.66	742.2	8.2	0.61	741.59	2.037	0.012	2.025	1.993	
2	23.2	296.36	740.8	8.0	0.59	740.21	2.041	0.015	2.026	1.989	
3	22.8	295.96	735.3	8.1	0.60	734.70	2.056	0.021	2.035	1.982	

Calculations:

T_m = 22.5 °C + 273.16 = 295.66 °K

Δp(mm Hg) = 0.0738 Δp(mm H_2O) = 0.0738 x 8.2 = 0.61 mm Hg

$P_m = P_B - \Delta p$ = 742.2 - 0.61 = 741.59 mm Hg

T_s = 298.16°K

P_s = 760 mm Hg

$V_m = V_f - V_i$ = 2.025 ℓ

*$V_s = P_m/P_s \times T_s/T_m \times V_m$ = 1.993 ℓ

$\bar{V}_s$ = Average of V_s values for the three tests = 1.987 ℓ

% Error = 100 $(\bar{V}_s - V_{st})/V_{st}$ = -.65 %, not to exceed ±1%

Figure 2.2. Wet test meter calibration log.

10. At the point at which the volumetric flask reaches its calibrated capacity, read the final volume (V_f) from the wet test meter dial and record.

11. Repeat steps 7 through 10 twice.

12. Calculate the volume at standard conditions, using equation 2-1, for each test.

$$V_s = \frac{P_m}{P_s} \times \frac{T_s}{T_m} \times V_m \qquad \text{Equation 2-1}$$

where

P_m = barometric pressure corrected for internal meter pressure, mm Hg

T_m = temperature of meter , °K(°K = °C + 273.16)

P_s = pressure at standard conditions = 760.0 mm Hg

T_s = temperature at standard conditions, °K (25 + 273.16 = 298.16°K)

V_m = volume as measured by wet test meter, ℓ

An example of this calculation is presented below:

a. Meter temperature = 22.5°C

b. Barometric pressure = 742.2 mm Hg

c. Wet test meter manometer reading = 8.2 mm

H_2O = 8.2 mm H_2O × 0.074 mm Hg/mm H_2O = 0.61 mm Hg

d. Volume as measured by wet test meter = 2.025 ℓ

e. Volume of volumetric flask = 2.0000 ℓ

$$P_m = (742.2 - 0.61) = 741.59 \text{ mm Hg}$$

$$T_m = (22.5 + 273.16) = 295.66 \text{ °K}$$

$$V_s = \frac{741.59}{760.00} \times \frac{298.16}{295.66} \times 2.025 = 1.993$$

Calculate the average volume at standard conditions by:

$$\overline{V}_s = \frac{V_{s1} + V_{s2} + V_{s3}}{3}$$

The relative percent error can be calculated as follows:

$$\% \text{ error} = \frac{\overline{V}_s - V_{st}}{V_{st}} \times 100$$

where V_{st} is the volume of the standard flask. An example computation using the above data is presented below:

$$\% \text{ error} = \frac{1.9870 - 2.0000}{2.0000} \times 100 = -0.65\%.$$

The error should not exceed ± 1%; should this error magnitude be exceeded, check all connections within the test apparatus for leaks and gravimetrically check the volume of the standard flask. Repeat the calibration procedure and if tolerance level is not met adjust the liquid level within the meter (see the manufacturer's manual) until the specifications are met.

2.1.2 Calibration of Soap Bubble Meter - Soap bubble meters calibrated by the manufacturer to an accuracy of ± 0.25% are commercially available. The volume of these soap bubble meter tubes are traceable to the National Bureau of Standards displacement volumes. Frequently, soap bubble meters are constructed in the laboratory employing a burette of an appropriate volume.

Two calibration methods are commonly employed for soap bubble meters, 1) volume displacement technique, as described for the wet test meter and 2) a method that employs measurement of the displacement volume using a gravimetric technique. Since the displacement volume of the soap bubble meter does not change with use, it requires only initial calibration upon receipt from the manufacturer. The soap bubble meter, when employed for calibration, can only be used over the entire volume for which it was calibrated. The gravimetric displacement method requires less effort. A calibration procedure is presented below.

1. Secure a clean displacement tube to a ring stand and attach a stopcock to the bottom as shown in Figure 2.3.
2. Obtain an appropriately sized, ground glass stoppered volumetric flask that will contain the volume of the displacement tube of the soap bubble meter.
3. Determine the weight of the flask and stopper to the following specifications:
    a. 10-ml flasks - weigh to the nearest 0.001 grams
    b. 100-ml flasks - weigh to the nearest 0.01 grams
    c. 1000-ml flasks - weigh to the nearest 0.1 grams

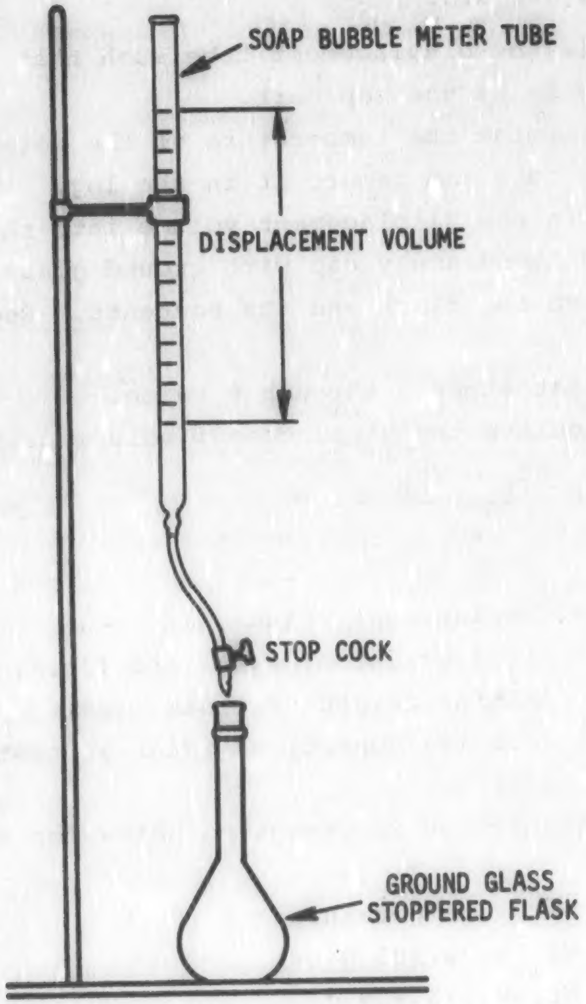
SOAP BUBBLE METER TUBE
DISPLACEMENT VOLUME
STOP COCK
GROUND GLASS STOPPERED FLASK

Figure 2.3 Test apparatus for soap bubble meter

Record the weight on the Soap Bubble Meter Calibration Log, Figure 2.4. A blank form for the use of the Handbook holder is given in Section 12.

4. Fill the displacement tube with distilled water and drain system until all air bubbles are removed. Collect this water in a beaker and discard.

5. Fill the displacement tube such that the bottom of the meniscus is at the top mark.

6. Determine the temperature of the water within the displacement tube and record it in the log.

7. Drain the displacement volume into the weighed flask (step 3) and immediately cap with ground glass stopper.

8. Weigh the flask and its contents. Record the weight in the log.

9. Repeat steps 3 through 8 twice.

10. Calculate the displacement volume using equation 2-2.

$$V_d = \frac{Wt_f - Wt_i}{D_r} \qquad \text{Equation 2-2}$$

where

V_d = displacement volume, ml
Wt_f = final weight of water and flask, grams
Wt_i = initial weight of flask, grams
D_r = relative density of water at test temperature, g/ml

An example calculation is presented below for a 100 ml displacement tube.

a. Water temperature = 23.0 °C
b. $Wt_i = 73.905$ g
c. $Wt_f = 173.930$ g
d. $D_r = 0.99756$. See Table 2.1, Relative density of water.
e. $V_d = \frac{173.930 - 73.905}{0.99756} = 100.27$ ml

11. Compute the average displacement volume $\bar{V}_d$ from the three tests,

Meter Serial No. 45324 Date 2/29/77

Displacement Volume 100 ml Calibrated by J. Doe

Test No.	Water Temperature °C	Wt_i g	Wt_f g	V_d ml	Remarks
1	23	73.905	173.930	100.27	
2	23	73.905	173.935	100.27	
3	23	73.905	173.925	100.26	

Calculations:

D_r = 0.99756 g/ml, from Table 2.1

$$V_d = \frac{Wt_f - Wt_i}{D_r} = \frac{173.93 - 73.905}{0.99756} = 100.27$$

$$\overline{V}_d = (V_{d_1} + V_{d_2} + V_{d_3})/3 = \frac{100.27 + 100.27 + 100.26}{3} = 100.267$$

V = Displacement volume stated by manufacturer = 100 ml

$$\% \text{ Error} = \frac{(\overline{V}_d - V) \times 100}{V} = 0.267$$, not to exceed 1%.

Figure 2.4 Soap bubble meter calibration log

Table 2.1 ABSOLUTE DENSITY OF WATER

The values presented below are numerically equal to the absolute density of H_2O in grams per milliliter (g/ml).

Temp °C	Density, g/ml	Temp, °C	Density, g/ml
15	0.99913	25	0.99707
16	0.99887	26	0.99681
17	0.99880	27	0.99654
18	0.99862	28	0.99626
19	0.99843	29	0.99597
20	0.99823	30	0.99567
21	0.99802	31	0.99537
22	0.99780	32	0.99505
23	0.99756	33	0.99473
24	0.99732	34	0.99440

$$\overline{V}_d = \frac{V_{d_1} + V_{d_2} + V_{d_3}}{3} \text{ ml.}$$

12. Compute the percent error as follows

$$\% \text{ error} = \frac{\overline{V}_d - V}{V} \times 100$$

where V = displacement volume as stated by the manufacturer (ml). An example calculation of % error assuming V = 100 ml and $\overline{V}_d$ is 100.267

$$\% \text{ error} = \frac{100.267 - 100}{100} \times 100 = 0.27\%.$$

The error should not exceed ± 1%.

The use of the soap bubble meter for the calibration of flow rate devices such as rotameters and mass flow meters requires correction for temperature, barometric pressure, and the vapor pressure of the soap bubble, which is considered to be that of the vapor pressure of water. Equation 2-3 is presented to assist the user of the soap bubble meter with respect to these corrections.

$$\text{Standard flow rate}^{a} = \frac{\text{Vol(ml)}}{\text{Time(min)}} \times \frac{(P - P_v)}{760} \times \frac{298.16}{(273 + T)} \qquad \text{Equation 2-3}$$

where

P = atmospheric pressure, mm Hg
P_v = vapor pressure of water, mm Hg (see Table 2.2)
T = temperature of gas, °C

Example: Assume temperature to be 20 °C and atmospheric pressure to be 710 mm Hg.

$$\text{Standard flow rate} = \frac{\text{Vol(ml)}}{\text{Time(min)}} \times \frac{(710 - 17.54)}{760} \times \frac{298.16}{(273.16 + 20)}$$

$$\underset{\text{(ml/min)}}{\text{Standard flow rate}} = \frac{\text{Vol(ml)}}{\text{Time(min)}} \times 0.927$$

---

[a] Standard conditions of 760 mm Hg, 25 °C, and 0% water vapor.

Table 2.2 SATURATION VAPOR PRESSURE OVER WATER (^oC, mm Hg)[a]

Values for Fractional Degree Between 50 and 89 Were Obtained By Interpolation

Temp. °C	0.0	0.2	0.4	0.6	0.8
−15	1.436	1.414	1.390	1.368	1.345
−14	1.560	1.534	1.511	1.483	1.460
−13	1.691	1.665	1.637	1.611	1.585
−12	1.834	1.804	1.776	1.748	1.720
−11	1.987	1.955	1.924	1.893	1.863
−10	2.149	2.116	2.084	2.050	2.018
− 9	2.326	2.289	2.254	2.219	2.184
− 8	2.514	2.475	2.437	2.399	2.362
− 7	2.715	2.674	2.633	2.593	2.553
− 6	2.931	2.887	2.843	2.800	2.757
− 5	3.163	3.115	3.069	3.022	2.976
− 4	3.410	3.359	3.309	3.259	3.211
− 3	3.673	3.620	3.567	3.514	3.461
− 2	3.956	3.898	3.841	3.785	3.730
− 1	4.258	4.196	4.135	4.075	4.016
− 0	4.579	4.513	4.448	4.385	4.320
0	4.579	4.647	4.715	4.785	4.855
1	4.926	4.998	5.070	5.144	5.219
2	5.294	5.370	5.447	5.525	5.605
3	5.685	5.766	5.848	5.931	6.015
4	6.101	6.187	6.274	6.363	6.453
5	6.543	6.635	6.728	6.822	6.917
6	7.013	7.111	7.209	7.309	7.411
7	7.513	7.617	7.722	7.828	7.936
8	8.045	8.155	8.267	8.380	8.494
9	8.609	8.727	8.845	8.965	9.086
10	9.209	9.333	9.458	9.585	9.714
11	9.844	9.976	10.109	10.244	10.380
12	10.518	10.658	10.799	10.941	11.085
13	11.231	11.379	11.528	11.680	11.833
14	11.987	12.144	12.302	12.462	12.624
15	12.788	12.953	13.121	13.290	13.461
16	13.634	13.809	13.987	14.166	14.347
17	14.530	14.715	14.903	15.092	15.284
18	15.477	15.673	15.871	16.071	16.272
19	16.477	16.685	16.894	17.105	17.319
20	17.535	17.753	17.974	18.197	18.422
21	18.650	18.880	19.113	19.349	19.587
22	19.827	20.070	20.316	20.565	20.815
23	21.068	21.324	21.583	21.845	22.110
24	22.377	22.648	22.922	23.198	23.476
25	23.756	24.039	24.326	24.617	24.912
26	25.209	25.509	25.812	26.117	26.426
27	26.739	27.055	27.374	27.696	28.021
28	28.349	28.680	29.015	29.354	29.697
29	30.043	30.392	30.745	31.102	31.461
30	31.824	32.191	32.561	32.934	33.312
31	33.695	34.082	34.471	34.864	35.261
32	35.663	36.068	36.477	36.891	37.308
33	37.729	38.155	38.584	39.018	39.457
34	39.898	40.344	40.796	41.251	41.710
35	42.175	42.644	43.117	43.595	44.078
36	44.563	45.054	45.549	46.050	46.556
37	47.067	47.582	48.102	48.627	49.157
38	49.692	50.231	50.774	51.323	51.879
39	52.442	53.009	53.580	54.156	54.737
40	55.324	55.91	56.51	57.11	57.72
41	58.34	58.96	59.58	60.22	60.86
42	61.50	62.14	62.80	63.46	64.12
43	64.80	65.48	66.16	66.86	67.56
44	68.26	68.97	69.69	70.41	71.14
45	71.88	72.62	73.36	74.12	74.88
46	75.65	76.43	77.21	78.00	78.80
47	79.60	80.41	81.23	82.05	82.87
48	83.71	84.56	85.42	86.28	87.14
49	88.02	38.90	89.79	90.69	91.59
50	92.51	93.5	94.4	95.3	96.3
51	97.20	98.2	99.1	100.1	101.1
52	102.09	103.1	104.1	105.1	106.2
53	107.20	108.2	109.3	110.4	111.4
54	112.51	113.6	114.7	115.8	116.9
55	118.04	119.1	120.3	121.5	122.6
56	123.80	125.0	126.2	127.4	128.6
57	129.82	131.0	132.3	133.5	134.7
58	136.08	137.3	138.5	139.9	141.2
59	142.60	143.9	145.2	146.6	148.0
60	149.38	150.7	152.1	153.5	155.0
61	156.43	157.8	159.3	160.8	162.3
62	163.77	165.2	166.8	168.3	169.8
63	171.38	172.9	174.5	176.1	177.7
64	179.31	180.9	182.5	184.2	185.8
65	187.54	189.2	190.9	192.6	194.3
66	196.09	197.8	199.5	201.3	203.1
67	204.96	206.8	208.6	210.5	212.3
68	214.17	216.0	218.0	219.9	221.8
69	223.73	225.7	227.7	229.7	231.7
70	233.7	235.7	237.7	239.7	241.8
71	243.9	246.0	248.2	250.3	252.4
72	254.6	256.8	259.0	261.2	263.4
73	265.7	268.0	270.2	272.6	274.8
74	277.2	279.4	281.8	284.2	286.6
75	289.1	291.5	294.0	296.4	298.8
76	301.4	303.8	306.4	308.9	311.4
77	314.1	316.6	319.2	322.0	324.6
78	327.3	330.0	332.8	335.6	338.2
79	341.0	343.8	346.6	349.4	352.2
80	355.1	358.0	361.0	363.8	366.8
81	369.7	372.6	375.6	378.8	381.8
82	384.9	388.0	391.2	394.4	397.4
83	400.6	403.8	407.0	410.2	413.6
84	416.8	420.2	423.6	426.8	430.2
85	433.6	437.0	440.4	444.0	447.5
86	450.9	454.4	458.0	461.6	465.2
87	468.7	472.4	476.0	479.8	483.4
88	487.1	491.0	494.7	498.5	502.2
89	506.1	510.0	513.9	517.8	521.8
90	525.76	529.77	533.80	537.86	541.95
91	546.05	550.18	554.35	558.53	562.75
92	566.99	571.26	575.55	579.87	584.22
93	588.60	593.00	597.43	601.89	606.38
94	610.90	615.44	620.01	624.61	629.24
95	633.90	638.59	643.30	648.05	652.82
96	657.62	662.45	667.31	672.20	677.12
97	682.07	687.04	692.05	697.10	702.17
98	707.27	712.40	717.56	722.75	727.98
99	733.24	738.53	743.85	749.20	754.58
100	760.00	765.45	770.93	776.44	782.00
101	787.57	793.18	798.82	804.50	810.21

[a] Handbook of Chemistry and Physics, 45th Edition, Chemical Rubber Publishing Company, 1965.

2.1.3 Calibration of Mass Flow Meters - Mass flow meters operate on a thermal principle that depends on the mass flow of the gas and its heat capacity to gauge the temperature within a heated conduit. Since these meters measure the true mass flow, they have the advantage of measuring flow rates without requiring corrections for changes of temperature and pressure. These values are usually given in standard cubic centimeters per minute, which is the volume occupied by a given mass of air at standard temperature and pressure as specified by the manufacturer. Since mass flow meters are not volume displacement devices, they require calibration, on at least a quarterly basis, against a displacement device such as a bubble meter or a wet test meter.

Mass flow meters can be calibrated as follows employing a calibrated wet test meter as a secondary calibration standard.[a]

1. With the mass flow meter off, adjust the meter to zero with the pointer needle adjustment screw located below the meter face.
2. Turn the meter on and allow it to warm up for one hour.
3. After the warm up period, adjust the electronic zero as follows:
   a. Plug the inlet and outlet of the transducer.
   b. Adjust electronic zero adjustment screw until the meter reads zero.
   c. Unplug the transducer and connect it to the test apparatus as illustrated in Figure 2.5.
4. Turn the vacuum pump on, and adjust the flow rate, with the needle valve, to approximately 75 percent of full scale of the mass flow meter.
5. Allow the system to equilibrate for approximately 10 revolutions of the wet test meter.
6. Read manometer on wet test meter in mm H_2O and convert to mm Hg (mm $H_2O \times 0.0738 =$ mm Hg), then record Δp on the Mass

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[a] Refer to the manufacturer's instructions for flow rate capacities.

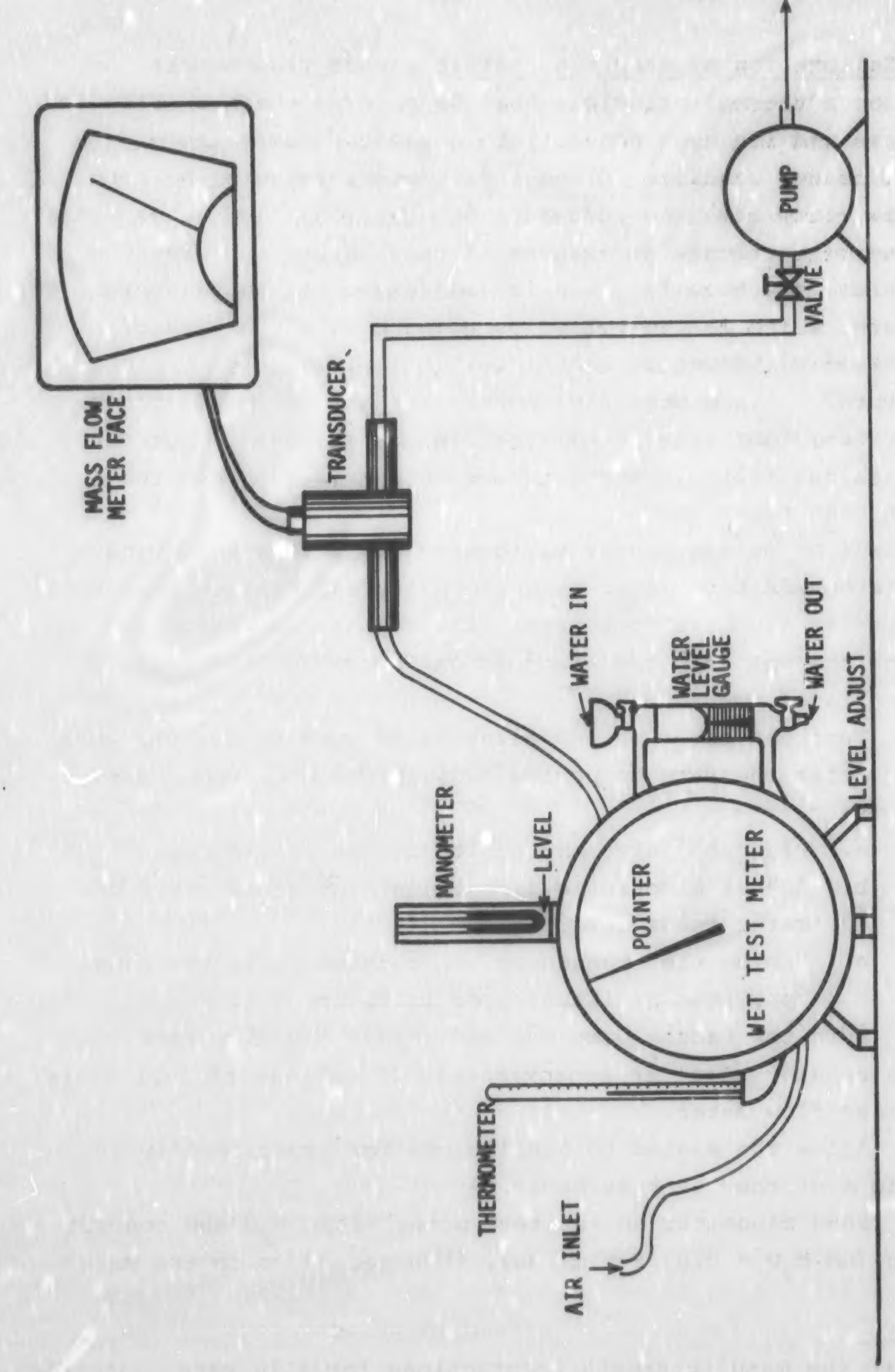

MASS FLOW METER FACE
TRANSDUCER
PUMP
VALVE
MANOMETER
LEVEL
POINTER
WET TEST METER
WATER IN
WATER LEVEL GAUGE
WATER OUT
LEVEL ADJUST
THERMOMETER
AIR INLET

Figure 2.5 Mass flow meter calibration apparatus

Flow Meter Calibration Log, Figure 2.6.

7. As the wet test meter pointer passes zero begin timing with a precision stopwatch. As the wet test meter pointer passes the three-quarter revolution mark read and record the mass flow meter reading in the log.

8. As the wet test meter pointer passes the starting point stop the stopwatch and record elapsed time, t.

9. Record the volume of the wet test meter in column headed by V_m.

10. Record wet test meter fluid temperature, T_m in °K.

11. Calculate V_s from P_m, T_m and V_m and record.

$P_s = 760$ mm Hg and $T_s = 298.16$.

$$V_s = \frac{P_m\ T_s}{P_s\ T_m} V_m.$$

12. Calculate flow rate Q_s from V_s and t and record.

$$Q_s = \frac{V_s}{t}.$$

13. Plot Q_s versus mass flow meter readings.

14. Repeat steps 6 through 13 for five other flow rates within the range of the mass flow meter.

15. Construct a best fit curve for the points generated, and use this relationship for future work employing the mass flow meter.

2.1.4 Calibration of SO_2 Sampling Train (Short Term Sampling) - The sampling train employed for sampling periods of one and three hours must be calibrated as it is used in the field. Two flow control systems are commonly employed. One uses a needle valve and the flow rate is determined by means of a rotameter. The other employs various size stainless steel hypodermic needles operated at critical flow conditions. A wet test meter is commonly used as the secondary standard to calibrate the flow rate within the system. An example of a calibration system for the rotameter flow control system is presented in Figure 2.7. A procedure for calibration of this system follows.

Mass Flow Meter Serial No. 47897 Calibrated with Wet Test Meter
Location laboratory Ambient/Room Temperature 295 °K
Barometric Pressure (P_B) 750 mm Hg Relative Humidity 50%
Calibrated by Joe Smith Date 3-13-75
mm Hg = 0.0738 × mm H_2O

Test Point	Wet Test Meter Readings				$V_s = \frac{P_m T_s}{P_s T_m} V_m$	Elapsed Time t (min)	$Q_s = \frac{V_s}{t}$ (ℓ/min)	Mass Flow Meter Scale
	Manometer Reading Δp (mm Hg)	$P_m = P_B - \Delta p$ P_m (mm Hg)	Meter Fluid Temperature T_m (°K)	Volume Displaced V_m (ℓ)				
1	0.738	749.262	295	1.0	0.9959	2.0	0.498	0.5
2								
3								
4								
5								
6								
7								
8								
9								
10								

Figure 2.6 Mass flow meter calibration log

NOTE: Rotameter and needle valve can be replaced with appropriate size hypodermic needle (see Section 2.1.10).

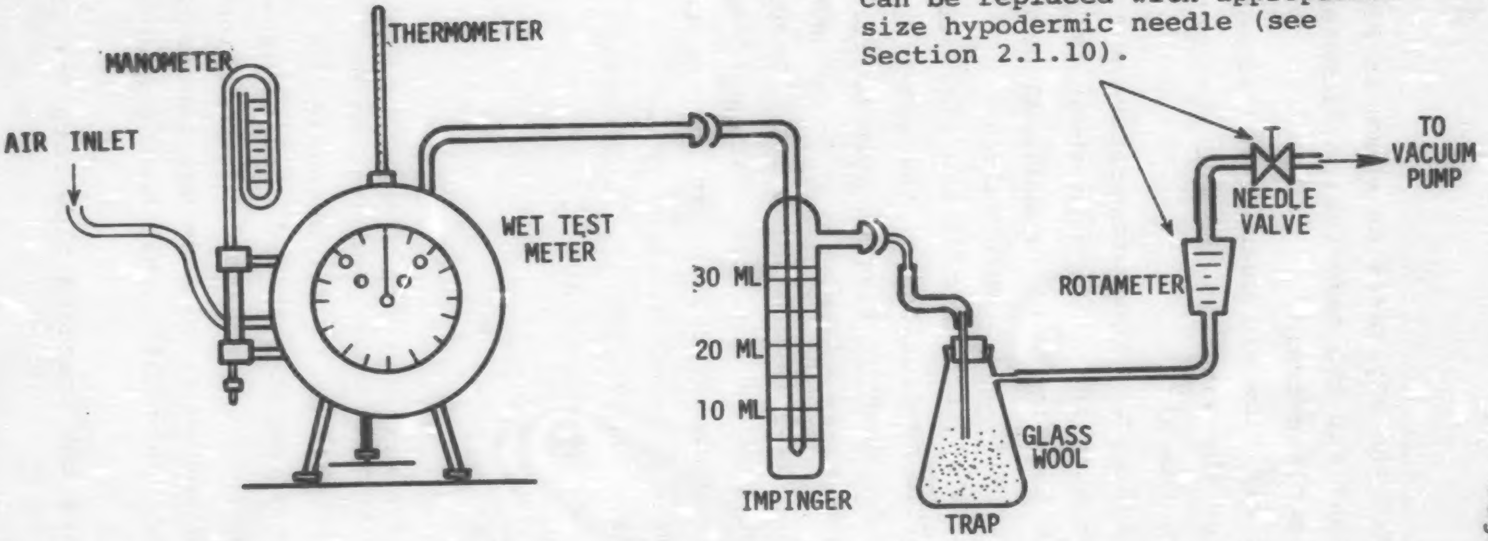

THERMOMETER
MANOMETER
AIR INLET
WET TEST METER
30 ML
20 ML
10 ML
IMPINGER
TRAP
GLASS WOOL
ROTAMETER
NEEDLE VALVE
TO VACUUM PUMP

Figure 2.7 Wet test meter calibration assembly for SO_2 sampling train

1. Prepare the apparatus as shown in Figure 2.7 using short connections and the same inside diameter tubing as used in the field sampling train.

2. Start air flow and operate for approximately 5-10 minutes to allow the water in the wet test meter to reach saturation with the air.

3. Read and record room temperature and barometric pressure on the sampling train data sheet (Figure 2.8).

4. Adjust the flow rate to about 20 percent of full scale of the rotameter with the needle valve.

5. With a stopwatch, record the time required to make at least two complete revolutions of the wet test meter. Repeat the above step for each of five or more uniformly spaced points on the rotameter scale, going from low values to high values. Repeat, going from high to low. Record rotameter reading, elapsed time of run, temperature of the liquid in the wet test meter, manometer reading at the wet test meter, and the total volume displaced for each run.

6. Calculate the volume (V_s) at standard conditions for each test point using equation 2-4,

$$V_s = \frac{P_m}{P_s} \times \frac{T_s}{T_m} \times V_m \qquad \text{Equation 2-4}$$

where

P_m = barometric pressure measured and corrected for internal meter pressure, mm Hg

T_m = temperature of air measured, °K

P_s = pressure at standard conditions = 760 mm Hg

T_s = temperature at standard conditions = 25°C = 298.16°K

V_m = volume measured from wet test meter, liters

An example of this calculation has been presented in Section 2.1.1, step 11.

7. Determine the standard flow rate for each test period as follows

$$Q_s = \frac{V_s}{t} = (\ell/\text{min}) \qquad \text{Equation 2-5}$$

Rotameter Serial No. 7774861 Calibrated with Wet Test Meter

Location laboratory Ambient/Room Temperature 22 °C + 273.16 = 295 °K

Barometric Pressure (P_B) 750 mm Hg Relative Humidity 50%

Calibrated by Joe Smith Date 3-14-75

Test Point	Rotameter Reading units	Elapsed Time t (min)	Temperature Liquid C°	Manometer Reading mm Hg[a]	Volume Displaced V_m (ℓ)	$V_s = \frac{P_m T_s}{P_s T_m} V_m$	$Q_s = \frac{V_s}{t}$ (ℓ/min)	Remarks
1	5.0	2.0	22	0.738	1.0	0.9959	0.498	
2								
3								
4								
5								
6								
7								
8								
9								
10								

[a] mm Hg = 0.0738 × mm H_2O

Figure 2.8 Rotameter calibration data sheet for SO_2 sampling train

where

V_s = volume of gas at standard conditions, liters
t = time, minutes

8. Plot the rotameter units versus the flow rate Q_s on linear graph paper, constructing a best fit smooth curve through the data points using a flexible rule. A typical calibration relationship is presented in Figure 2.9. All data points should be within ± 2% of the curve of best fit.

9. Should the sampling train be employed in a field location where the barometric pressure and/or temperature is different than those conditions when the rotameter was calibrated, the following corrections must be applied to convert the flow rate to standard conditions.

$$Q_s = Q_f\left(\frac{P_f}{760} \times \frac{298.16}{T_f}\right)^{1/2} \qquad \text{Equation 2-6}$$

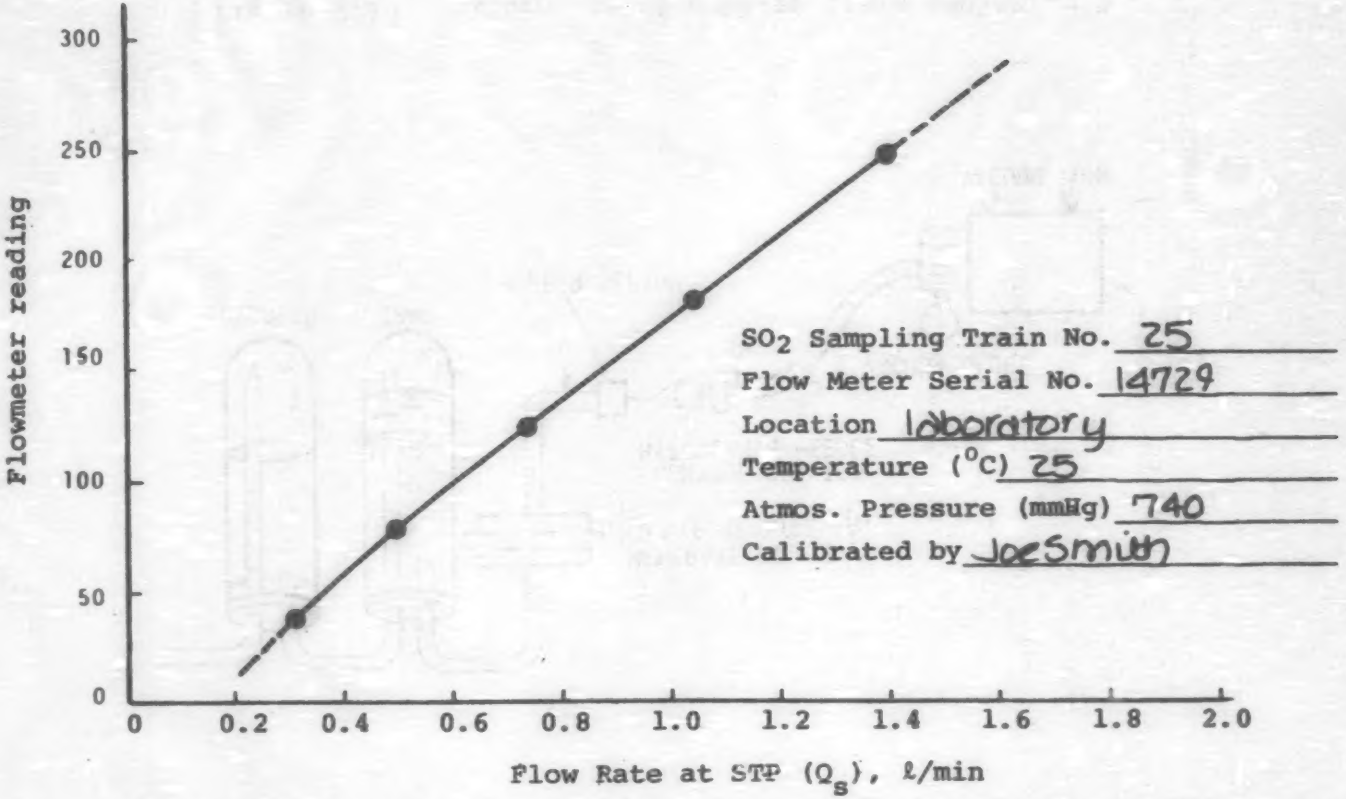
where

Q_s = flow rate corrected to standard conditions from field conditions, ℓ/min
Q_f = flow rate at field conditions from calibration curve, ℓ/min
P_f = barometric pressure at field conditions, mm Hg
T_f = temperature at field conditions, average temperature of sampling train, °K.

2.1.5 Calibration of SO_2 Sampling Train - SO_2 sampling trains used for collecting 24-hour SO_2 samples differ from the short-term SO_2 sampling train with respect to the type of bubbler used and in the method of flow control and measurement. Figure 2.10 is an example of a 24-hour SO_2 sampling train employing a critical orifice. In this sampling train a 3/8 inch, 27-gauge hypodermic needle[a] is used as a critical orifice and serves as an inexpensive air flow control device. When operated at critical flow conditions this method will produce a constant flow rate in the range of 0.2 ℓ/min. Prior to use, the sampling train should be leak checked. The hypodermic needle must be calibrated before use. Recalibration of the needle after the

---

[a] Other types of critical orifices in addition to hypodermic needles are also used.



SO_2 Sampling Train No. 25

Flow Meter Serial No. 14729

Location laboratory

Temperature (^oC) 25

Atmos. Pressure (mmHg) 740

Calibrated by Joe Smith

Figure 2.9 Typical rotameter calibration curve for SO_2 sampling train

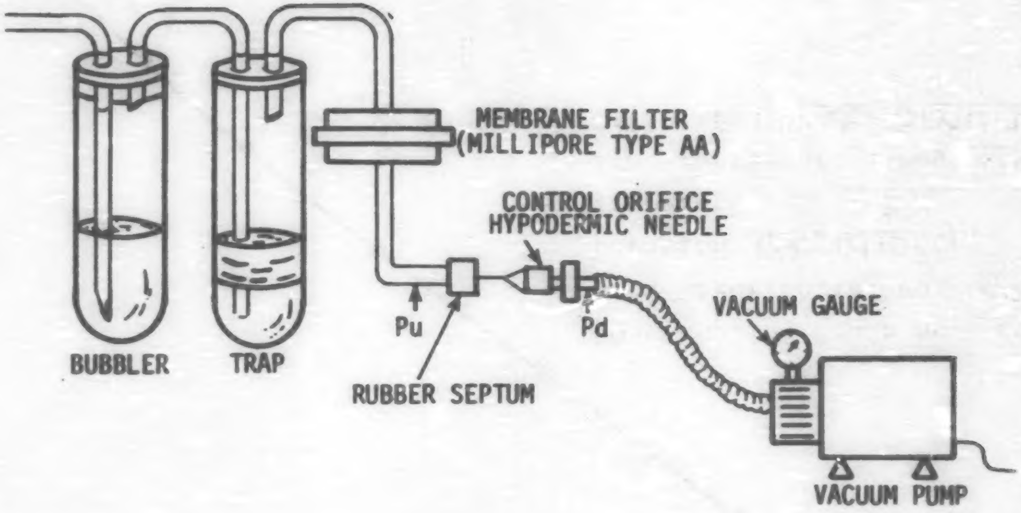

MEMBRANE FILTER
(MILLIPORE TYPE AA)
CONTROL ORIFICE
HYPODERMIC NEEDLE
VACUUM GAUGE
Pu
Pd
BUBBLER
TRAP
RUBBER SEPTUM
VACUUM PUMP

Figure 2.10 Calibration of bubbler train employing a critical orifice

sampling period must also be performed to assure that there has been no change in flow rate during sampling. Mass flow meters, rotameters, and bubble meters can be used for calibrating the hypodermic needle. The wet test meters are not recommended because they require considerably more time than the other methods.

Although the critical flow hypodermic needle is used mainly in the 24-hour SO_2 sampling train, it may also be used in the short term sampling trains if larger needle sizes are used. Typical needle sizes and their flow rate under critical conditions, are presented below.

1. 30-minute samples, 1-inch, 22-gauge hypodermic needle, will provide a sampling rate of about 1 ℓ/min.
2. 1-hour samples, 5/8 inch, 23-gauge hypodermic needle, will provide a sampling rate of about 0.5 ℓ/min.
3. 24-hour samples, 3/8 inch, 27-gauge hypodermic needle, will provide a sampling rate of about 0.2 ℓ/min.

Constant flow rates through hypodermic needles can only be achieved when critical flow conditions are met. These conditions are specified as being when the absolute pressure downstream of the needle (P_d) is less than or equal to 0.45 of the absolute pressure upstream of the needle (P_u), i.e., $P_d \leq 0.45\ P_u$.[a] For the 24-hour sampling train as shown in Figure 2.10, the upstream pressure (P_u) is usually 25 mm Hg less than the barometric pressure (P_b), and the downstream pressure (P_d) is equal to the barometric pressure minus the pressure from the vacuum gauge (P_g), i.e., $P_d = P_b - P_g$. By substitution

$$P_d \leq 0.45\ P_u$$

implies

$$P_b - P_g \leq 0.45\ (P_b - 25)$$

or

$$P_g \geq 0.55\ P_b + 11.25.$$

---

[a] Reference 8 of the Reference Method, Section 2.1.10, verifies the value 0.45 for use with the hypodermic needle. Reference 5 of Section 2.1.11 gives the value 0.53 as a purely theoretical value.

Assuming that calibration is carried out at a barometric pressure of 760 mm Hg, then

$P_g \geq 429.25$ mm Hg or 16.9 in.Hg.

If the vacuum gauge in Figure 2.10 reads 16.9 in. Hg or greater, then critical flow conditions for the 24-hour SO_2 sampling train will be met. Since the barometric pressure decreases with altitude, critical flow conditions will be satisfied at lower vacuum gauge readings at higher altitudes. Some vacuum gauge readings for critical flow at several altitudes are shown in Table 2.3. A minimum safety factor of 20 percent of the gauge reading should be added to ensure critical flow during the total sampling period.

Table 2.3 VACUUM GAUGE READINGS TO ACHIEVE CRITICAL FLOW FOR VARIOUS ELEVATIONS
(Pressure Drop Across Sampling Train Assumed to be 25 mm Hg)

Elevation, feet above sea level	Standard barometric pressure		Gauge reading for critical flow	
	mm Hg	in.Hg	mm Hg	in.Hg
0	760	29.92	429	16.89
1000	732	28.82	414	16.29
2000	706	27.78	399	15.72
3000	680	26.77	385	15.16
4000	656	25.81	372	14.63
5000	632	24.87	359	14.12
6000	609	23.98	346	13.63

Flow rate through hypodermic needles used as critical orifices are directly affected by the upstream pressure, which in turn is affected by the barometric pressure. Care must be exercised when needles are used at elevations that differ greatly from that at which they were calibrated. Corrections can be made with the following equation,

$$Q_s = Q_f\left(\frac{P_f}{P_c}\right)^{1/2}$$, T (°K) is constant,

where

Q_f = flow rate at field conditions from calibration curve
Q_s = flow rate corrected to standard conditions
P_f = barometric pressure during field sampling
P_c = barometric pressure during calibration.

2.2 Calibration of Critical Flow Orifices

Three stepwise procedures are presented below for the calibration of critical flow orifices employing mass flow meter, soap bubble meter, and rotameter techniques.

2.2.1 Critical Orifice Calibration with Mass Flow Meter -

1. Assemble the equipment as shown in Figure 2.11.

2. Place a new serum stopper on tube.

3. Adjust the water level in the sample tube if necessary.

4. Assure tightness of all connections.

5. Assure line is connected to the mass flow meter transducer outlet and to the sample tube impinger side.

6. Turn the vacuum pump ON and check the vacuum by placing a finger over the end of the tube. The vacuum gauge should read at least 20 inches of Hg at a barometric pressure of 760 mm. Turn pump OFF.

7. Zero the mass flow meter.

8. Insert a needle through the septum center perpendicular to the plane of the face of the septum.

9. Turn the vacuum pump ON.

10. Slide needle holder snugly into the recess at the base of the needle. Check for bubbles in the sample tube.

11. Read flow rate from the mass flow meter after meter indicator is stabilized (this stabilization period requires approximately 30 seconds). Read vacuum gauge to make sure vacuum is critical during flow. Shut off pump.

12. Turn on pump and repeat step 11 two more times.

13. Determine average of the three flow rates.

14. For 24-hour samples, if average flow rate is less than 180 ml/min or exceeds 220 ml/min needle should be discarded.

15. If needle flow rate is between 180 to 220 ml/min, assign it a number, record the number on the needle with an appropriate ink pen, record the number and flow rate in critical orifice calibration log, Figure 2.12. Place needle in protective container and store until needed.

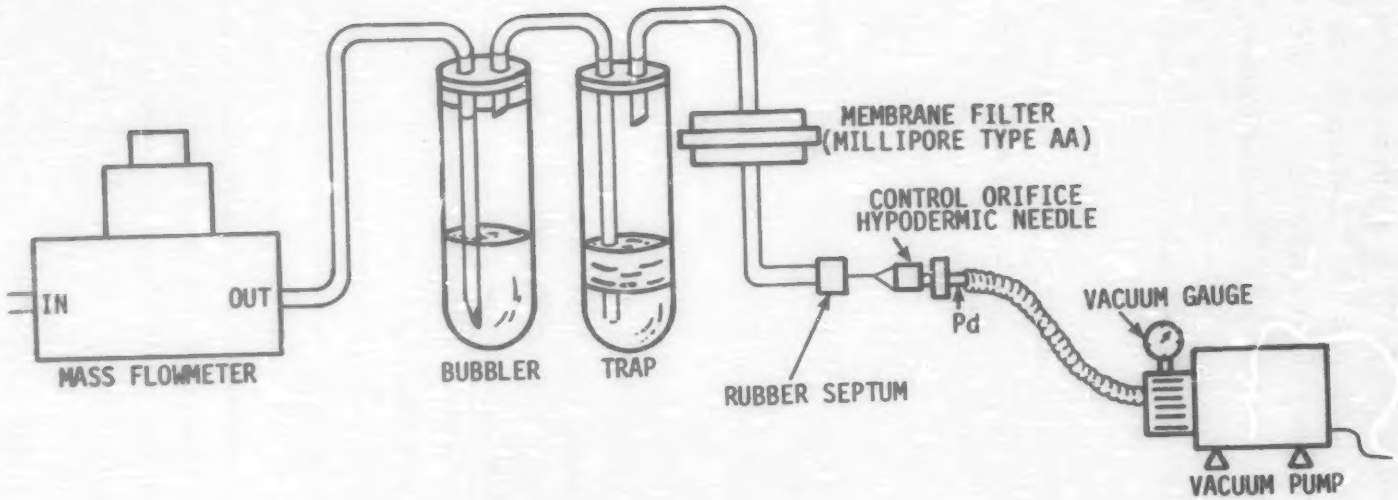

MEMBRANE FILTER
(MILLIPORE TYPE AA)
CONTROL ORIFICE
HYPODERMIC NEEDLE
IN
OUT
MASS FLOWMETER
BUBBLER
TRAP
RUBBER SEPTUM
Pd
VACUUM GAUGE
VACUUM PUMP

Figure 2.11. Calibration of critical orifice and bubbler train employing mass flow meter

Needle Identification Number	Date of Calibration	Calibration Method	Flow rate at STP ml/min	Analyst	Remarks
28	5-26-75	Mass Flow Meter	200	g.o.	

Figure 2.12 Calibration log for critical flow orifices

2.2.2 Critical Orifice Calibration with Bubble Meter -

1. Assemble the equipment as shown in Figure 2.13.

2. Place a new serum stopper on the tube.

3. Adjust the water level in the sample tube if necessary.

4. Assure tightness of all connections.

5. Assure line is connected to the bubble meter and to the sample tube impinger side.

6. Turn the vacuum pump ON and check the vacuum by placing a finger over the end of the tube. The vacuum gauge should read at least 20 inches of Hg at a barometric pressure of 760 mm. Turn the vacuum pump OFF.

7. Insert a needle through the septum center perpendicular to the plane of the face of the septum.

8. Turn the vacuum pump ON.

9. Slide needle holder snugly into the recess at the base of the needle. Check for bubbles in the sampling tube.

10. Touch surface of soap solution with open end of bubble meter so that a soap bubble will start to travel up the bubble meter tube. Repeat several times or until bubbles will travel full length of tube without breaking.

11. Touch surface of soap solution with open end of bubble meter so that a single bubble starts to travel up the bubble tube. When bubble passes first line of graduated scale, start stopwatch. Record time and volume displaced. Read vacuum gauge to make sure vacuum is critical during flow.

12. Repeat step 11 two more times. Record time and volume each time.

13. Calculate average time and volume displacement for three runs.

14. Correct average volume displacement to standard conditions of 760 mm Hg and 25 °C by equation 2-7,

$$V_s = V_m \frac{(P - P_v)}{P_s} \times \frac{T_s}{T_m} \qquad \text{Equation 2-7}$$

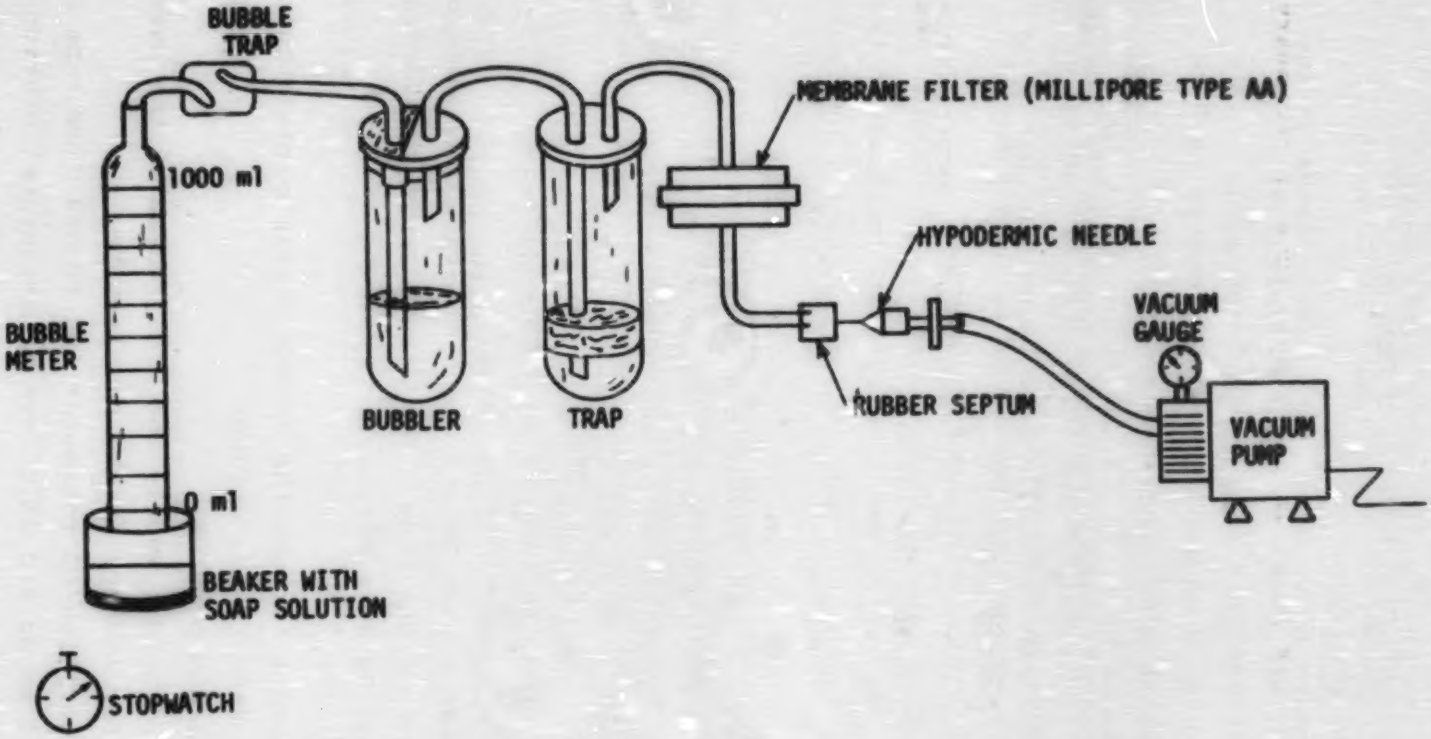

BUBBLE TRAP
MEMBRANE FILTER (MILLIPORE TYPE AA)
1000 ml
HYPODERMIC NEEDLE
VACUUM GAUGE
BUBBLE METER
BUBBLER
TRAP
RUBBER SEPTUM
VACUUM PUMP
0 ml
BEAKER WITH SOAP SOLUTION
STOPWATCH

Figure 2.13. Calibration of critical orifice and bubbler train employing a soap bubble meter

where

V_m = measured volume from bubbler meter, ml

P = atmospheric pressure, mm Hg

P_v = vapor pressure of water in soap bubble meter at the temperature employed, mm Hg (see Table 2.2)

P_s = pressure at standard conditions = 760 mm Hg

T_s = temperature at standard conditions, 298.16 °K

T_m = temperature of soap bubble meter, °K.

15. Divide corrected average volume displacement by average time to determine the flow rate.

16. If flow rate exceeds 220 ml/min or is less than 180 ml/min needle should be discarded and destroyed.

17. If needle flow rate is between 180 to 220 ml/min, assign it a number, record the number and flow rate in critical orifice calibration log, place needle in protective container, and store until needed.

2.2.3 Critical Orifice Calibration with Rotameter -

1. Assemble the equipment as shown in Figure 2.14.
2. Place a new serum stopper on the tube.
3. Adjust the water level in the sampling tube if necessary.
4. Assure tightness of all connections.
5. Assure the line from the surge tank outlet is connected to the sample tube impinger side.
6. Turn the vacuum pump ON and check the vacuum by placing a finger over the end of the tube. The vacuum gauge should read at least 20 inches of Hg at a barometric pressure of 760 mm. Turn the vacuum pump OFF.
7. Insert a needle through the septum center perpendicular to the plane of the face of the septum.
8. Turn the vacuum pump ON.
9. Slide needle holder snugly into the recess at the base of the needle. Check for bubbles in the sample tube.
10. Read ball position on rotameter and record flow rate corrected to STP of 760 mm Hg and 25°C from rotameter calibration curve. Read vacuum gauge to make sure vacuum is critical during flow.

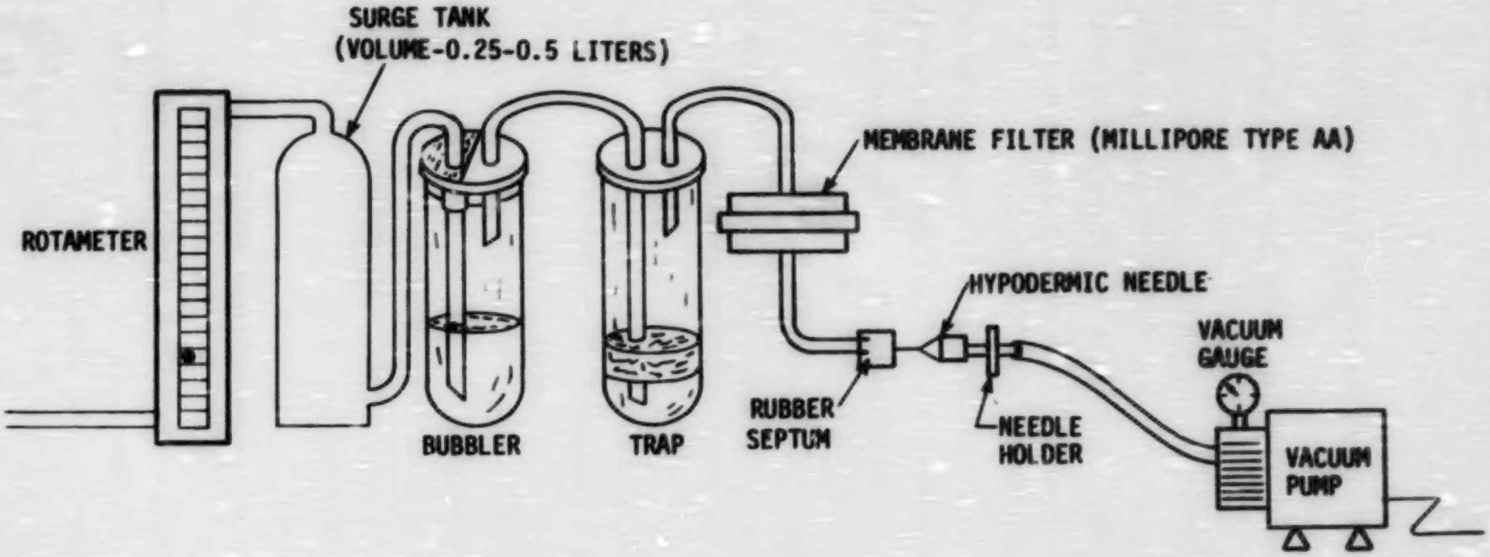

SURGE TANK
(VOLUME-0.25-0.5 LITERS)
MEMBRANE FILTER (MILLIPORE TYPE AA)
ROTAMETER
HYPODERMIC NEEDLE
VACUUM
GAUGE
RUBBER
SEPTUM
NEEDLE
HOLDER
BUBBLER
TRAP
VACUUM
PUMP

Figure 2.14. Calibration of critical orifice and bubbler employing a rotameter

11. Repeat step 10 two more times. Turn pump OFF then back ON after each run.

12. Calculate the average flow rate for the three runs.

13. Should the critical orifice be employed in a field location where the barometric pressure and/or temperature is different from those conditions when the rotameter was calibrated, use equation 2-6 to convert the flow rate to standard conditions.

14. If flow rate exceeds 220 ml/min or is less than 180 ml/min needle should be discarded and destroyed.

15. If needle flow rate is between 180 to 220 ml/min assign it a number, record the number and flow rate in the critical orifice calibration log, place needle in protective container and store until needed.

2.3 Calibration of Analytical Balance

The balance calibration should be verified when the balance is 1) first purchased, 2) any time the balance has been moved or subjected to rough handling, or 3) during routine operations when a standard weight cannot be weighed within ± 0.5 mg of its stated weight. An example of a typical balance performance log is presented in Figure 2.15. If at any time one or more of the standard weights cannot be measured within ± 0.5 mg of its stated value, have the balance recalibrated. The manufacturer should perform the calibration and subsequent adjustments. Record the results of all balance checks in the log book, see Figure 2.15.

2.4 Calibration of Elapsed Time Meter

The elapsed time meter (synchronous motor type - 60 hertz) should be checked on site or in the laboratory every six months against a timepiece of known accuracy. If the indicator shows any signs of being temperature sensitive, it should be checked on site during each season of the year.

A gain or loss of more than 2 minutes in a 24-hour period warrants an adjustment or replacement of the indicator.

Record results of these checks in the calibration log book.

ANALYTICAL BALANCE QUALITY CONTROL LOG

Date	Time	Class "S" Weights (g)			Technician
		0.5000	1.0000	2.0000	
7/29/74	11:07	0.5000	1.0002	2.0000	BSM
7/29/74	12:08	0.5002	1.0003	2.0001	DEK
7/29/74	2:40	0.5000	1.0000	1.9999	DEK
7/30/74	4:03	0.4996	0.9999	2.0002	JLK
7/31/74	9:57	0.4994	1.0000	2.0000	DEK
7/31/74	10:56	0.4995	0.9992	1.9993	DEK
7/31/74	11:57	0.4994	0.9994	1.9994	DEK
7/31/74	2:04	0.5001	1.0000	2.0002	DEK
7/31/74	3:05	0.5000	1.0000	2.0000	DEK
8/1/74	9:03	0.4998	0.9992	1.9992	DEK
8/1/74	10:05	0.4993	0.9992	1.9992	DEK
8/1/74	11:10	0.5000	1.0001	2.0001	DEK
8/1/74	12:12	0.4998	0.9997	1.9993	DEK
8/1/74	1:43	0.5000	1.0001	2.0002	DEK
8/1/74	2:42	0.5001	1.0001	2.0001	DEK
8/1/74	3:45	0.5001	1.0000	2.0001	DEK
8/2/74	8:54	0.5000	1.0001	2.0001	DEK
8/2/74	9:56	0.5000	1.0000	2.0001	DEK
8/2/74	10:59	0.5003	0.9999	1.9998	DEK
8/2/74	12:16	0.5001	1.0002	2.0002	DEK
8/2/74	1:55	0.4990	1.0002	2.0001	DEK
8/2/74	3:03	0.5000	0.9999	2.0001	DEK
8/2/74	4:00	0.5000	0.9998	1.9999	DEK
8/3/74	8:41	0.4999	0.9996	1.9998	DEK
8/3/74	11:15	0.5002	1.0002	2.0002	DEK
8/5/74	8:42	0.5001	1.0000	2.0000	DEK
8/5/74	9:45	0.5000	1.0000	2.0000	DEK
8/5/74	10:44	0.5000	1.0000	2.0000	DEK
8/5/74	11:46	0.5000	1.0001	2.0000	DEK
8/5/74	1:16	0.5001	1.0000	2.0000	DEK
8/5/74	2:21	0.5001	1.0000	2.0001	DEK
8/5/74	3:15	0.5000	1.0000	2.0001	DEK
8/6/74	9:37	0.4999	1.0000	2.0000	DEK
8/6/74	11:05	0.5000	0.9998	1.9994	DEK
8/6/74	12:10	0.4999	0.9998	1.9999	DEK
8/6/74	2:10	0.5000	0.9999	2.0000	DEK
8/6/74	3:09	0.5000	1.0000	2.0000	DEK
8/6/74	4:05	0.5000	1.0000	2.0000	DEK
8/7/74	8:50	0.5000	1.0002	2.0003	DEK
8/7/74	9:46	0.4996	0.9992	1.9990	DEK
8/7/74	1:10	0.5001	1.0000	2.0000	DEK
8/7/74	2:20	0.5001	1.0000	2.0000	DEK
8/7/74	3:25	0.5002	1.0001	2.0000	DEK
8/8/74	9:46	0.5000	1.0000	2.0000	DEK
9/24/74	3:50	0.5001	1.0001	2.0001	DEK
9/26/74	3:01	0.5001	1.0001	2.0001	DEK

Figure 2.15 Example of balance performance record

2.5 Calibration of ON-OFF Timer

For those samplers that are equipped with an on-off timer, the timer should be calibrated and adjusted using a calibrated elapsed time meter as the reference. An example of this type calibration procedure is presented below.[6] Figure 2.16 depicts the wiring diagram for use in this calibration.

The timer calibration procedure should be performed on a quarterly basis. Calibration data are recorded in the Timer Calibration Log. See Figure 2.17 for an example and Section 2.1.12 for a blank form for the Handbook user.

1. Plug a correctly wired timer into an electrical outlet.
2. Set the timer to the correct time.
3. Set the ON and OFF time trippers for a 24-hour test period.
4. Plug the test light into one of the output plugs and an elapsed time meter into the other.
5. Check the system by manually operating the switch ON and OFF.
6. Allow the system to operate for the 24-hour test period and detemine the elapsed time from the elapsed time meter.
    a. If the elapsed time is 24 hours ± 15 minutes, the timer is acceptable for field use.
    b. If the elapsed time is not 24 hours ± 15 minutes, adjust the tripper switches and repeat the test.

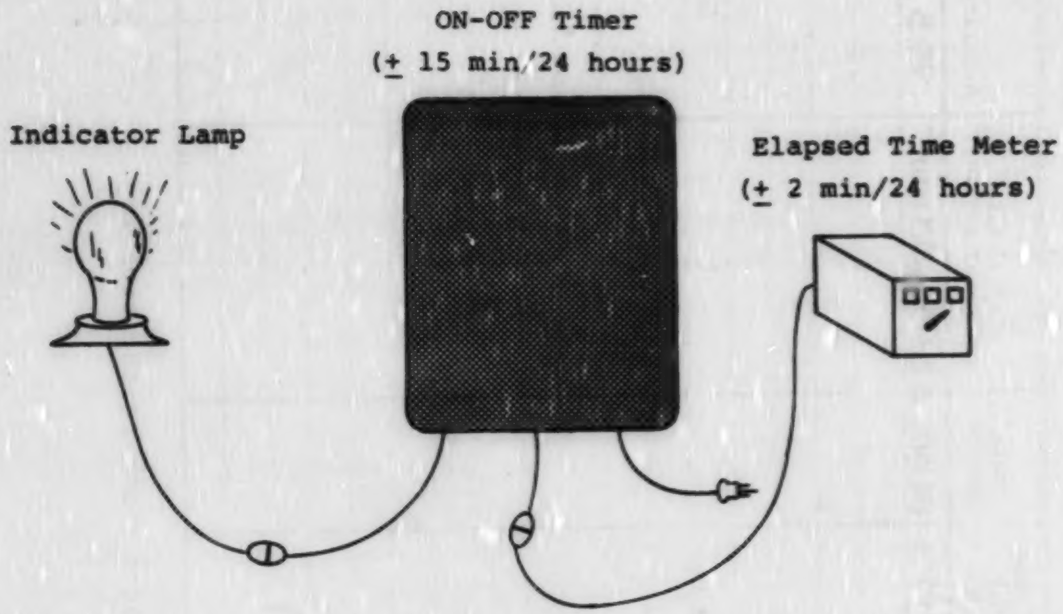

ON-OFF Timer
(± 15 min/24 hours)
Indicator Lamp
Elapsed Time Meter
(± 2 min/24 hours)

Figure 2.16 Diagram of a timer calibration system

Site location	Timer No.	Date		Elapsed time indicator serial No.	Test period	Elapsed time indicator reading	Accuracy	By (signature)
		Start	Stop					
1141	4321	6/10/75	6/11/75	CW080G	24 hrs.	23hrs. 53min.	99.5	C. Jones

Figure 2.17 Example of a timer calibration log

Table 2.4 ACTIVITY MATRIX FOR CALIBRATION OF EQUIPMENT

Equipment	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Wet test meter	Percent error not to exceed 1%, see Figure 2.2	Checked upon receipt and quarterly thereafter with liquid positive displacement technique, see Section 2.1.1	Check connections, gravimetrically check the volume of the standard flask, repeat calibration and if limits are met, adjust meter as per manufacturer's instructions
Soap bubble meter	Percent error not to exceed 1%, see Figure 2.4	Initial calibration upon receipt from manufacturer, gravimetric displacement method, Section 2.1.2	Adjust per manufacturer's instructions or return to manufacturer
Mass flow meter	All data points within ±2% of the best-fit curve	Calibrate vs. bubble or wet test meter (quarterly), see Section 2.1.3	As above
Rotameter	As above	Before each field use and after sampling calibrate vs. wet meter, see Section 2.1.4	As above
Critical orifice	24-hr sample:Flow rate between 180 and 220 ml/min 1-hr:Flow rate between 450 and 550 ml/min 1/2-hr:Flow rate between 900 and 1100 ml/min	Before use, and after sampling calibrate vs. mass flow meter, (Sec. 2.2.1), or vs. bubble meter (Sec. 2.2.2) or vs. rotameter (Sec. 2.2.3)	Destroy and discard needle
Analytical balance	Standard weights within 0.5 mg of their stated weight	First purchased, after moving or subjection to rough handling, or when a standard weight cannot be weighed within 0.5 mg, use standard weight	Repeat check to verify malfunction. Manufacturer should perform the recalibration and adjustment as required
Elapsed time meter	±2 minutes/24-hours	Check every 6 months against timepiece of known accuracy	Adjustment or replacement
ON-OFF timer	±15 minutes/24-hours	Perform quarterly using calibrated elapsed time meter, Section 2.5	Adjust the tripper switches and repeat test

3.0 PREPARATION OF REAGENTS

A summary of the quality control functions for preparation of reagents is contained in Table 3.1 at the end of this section.

3.1 Distilled Water

The standard specification for reagent water is described in the ASTM Designation: D 1193-72, pages 203 to 205. This document requires Type II grade of water which is prepared by distillation using a still designed to produce a distillate having a conductivity of less than 1.0 micromho/cm at 25°C and pH range of 6.6 to 7.2. Perform the following purity test for reagent water after each lot of reagents is prepared only if the slope and intercept of the standard curve fail to meet the specified criteria (see Section 2.1.5).

1. Add 0.20 ml of $KMnO_4$ solution (0.316 g/liter) to a mixture of 500 ml of the distilled water and 1 ml of H_2SO_4 in a stoppered bottle of chemically resistant glass.

2. If the permanganate color (blue) does not disappear completely after standing for 1 hour at room temperature, consider the water suitable for use.

3. If the permanganate does disappear, the water must be purified before using.

4. Water failing the purity test can be purified as follows:
   a. Add one gram each of potassium permanganate and barium hydroxide for each liter of distilled water.
   b. Redistill the water in an all-glass still.
   c. Perform the test for purity as described above.

5. Repeat the purification procedure and the test for purity until slope and intercept of the standard curve meet the specified limits.

6. Store reagent water in inert containers with vent guard. (A suitable vent guard is a drying tube filled with equal parts of 8-20-mesh soda lime, oxalic acid, and 4-8-mesh calcium chloride, each compound being separated from the other by a glass wool plug. Replace vent guard drying materials every 30 days.)

3.2 Absorbing Reagent

3.2.1 Absorbing Reagent Preparation - Prepare absorbing reagent (0.04 M potassium tetrachloromercurate (TCM):

1. Dissolve 10.87 g mercuric chloride, 0.066 g EDTA (ethylenediaminetetraacetic acid, disodium salt) and 6.0 g potassium chloride in distilled water and dilute to one liter. Usually it is necessary to prepare larger quantities of absorbing reagent to service the sampling requirements of the agency. WARNING: Highly poisonous and caustic. If spilled on skin, flush off with water immediately. Rubber gloves should be worn when preparing absorbing reagent.

2. Label reagent bottle with date of preparation.

3.2.2 Absorbing Reagent pH Test - For each lot of absorbers prepared for sampling:

1. Standardize pH meter with pH of 4.0 and 7.0 buffer solutions.

2. Measure pH of absorbing reagent: optimal pH 4.0.

3. Discard reagent if $pH < 3.0$ or $pH > 5.0$.

3.2.3 Absorbing Reagent Storage - Store absorbing reagent in stoppered container. Discard if reagent is more than 6 months old. (Reagent is normally stable for 6 months.) Examine visually before each use; discard if visible precipitate has formed.

3.3 Sulfamic Acid (0.6 percent)

Dissolve 0.6 g sulfamic acid in a 100-ml volumetric flask with water, and bring to mark. Keep in a glass-stoppered flask while not in use. Prepare fresh daily.

3.4 Formaldehyde (0.2 percent)

Dilute 5 ml formaldehyde solution (36-38 percent) to 1000 ml with distilled water. Keep in a stoppered container while not in use. Prepare fresh daily.

3.5 Starch Indicator Solution

Triturate 0.4 g soluble starch and 0.002 g mercuric iodide (preservative) with a little water, and add paste slowly to 200 ml boiling water. Continue boiling until solution is clear; cool and transfer to a glass-stoppered bottle. Alternatively, a solution of stabilized starch for iodimetric determinations can be purchased commercially.

3.6 Stock Iodine Solution (0.1 N)

Place 12.7 g iodine in a 250 ml beaker; add 40 g potassium iodine and 25 ml of water. Stir until all is dissolved, then transfer to a 1000-ml flask and dilute to the mark with distilled water. Keep the solution in a glass-stoppered, dark bottle and store in a cool place.

3.7 Working Iodine Solution (0.01 N)

Prepare approximately 0.01 N iodine solution by diluting 50 ml of stock solution to 500 ml with distilled water. Keep in a glass-stoppered, dark bottle or flask. Prepare fresh daily.

3.8 Stock Sodium Thiosulfate Solution (0.1 N)

Prepare a stock solution by dissolving 25 g sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in 100 ml freshly boiled, cooled distilled water and add 0.1 g sodium carbonate to the solution Allow the solution to stand 1 day before standardizing. Keep in glass-stoppered flask. Standardize against potassium iodate standard before each use. To standardize, accurately weigh to the nearest 0.1 mg, 1.5 g ACS Reagent grade potassium iodate (KIO_3) dried at 180°C for 3 hours in a drying oven and cooled in a desiccator. Dilute to volume in a 500 ml volumetric flask. To a 500-ml iodine flask, pipette 50 ml of iodate solution, add 2 g potassium iodide and 10 ml of 1 N hydrochloric acid. Stopper the flask. After 5 minutes, titrate with stock thiosulfate solution to a pale yellow. Add 5 ml starch indicator solution and continue the titration until the blue color disappears.

Calculate the normality by:

$$N = \frac{W}{M} \times 2.80$$

where

N = normality of stock thiosulfate solution

M = volume of thiosulfate required, ml

W = weight of potassium iodate, grams

$$2.80 = \frac{10^3 \text{ (conversion of g to mg)} \times 0.1 \text{ (fraction iodate used)}}{35.67 \text{ (equivalent weight of potassium iodate)}}$$

3.9 Sodium Thiosulfate Titrant (0.01 N)

Accurately pipette 100 ml of the stock thiosulfate solution into a 1000 ml volumetric flask. Dilute to the mark with freshly boiled, cooled distilled water. This 0.01 N solution is not stable, and must be prepared fresh daily from the stock thiosulfate solution. Keep in a glass-stoppered flask or bottle when not in use.

3.10 Standard Sulfite-TCM Solution

Dissolve 0.3 g sodium metabisulfite ($Na_2S_2O_5$) or 0.4 g sodium sulfite (Na_2SO_3) in 500 ml of recently boiled, cooled, distilled water. (Sulfite solution is unstable; it is important, therefore, to use water of the highest purity to minimize this instability.) This solution contains the equivalent of 320 to 400 μg/ml of SO_2. The actual concentration of the solution is determined by adding excess iodine and back-titrating with standard sodium thiosulfate solution. To standardize the sulfite solution, pipette 50 ml of the 0.01 iodine into each of six 500-ml iodine flasks,with three flasks labelled A and three flasks labelled B. To each flask A (blank) add 25 ml of distilled water, and to each flask B (sample) pipette 25 ml of standard sulfite solution. Stopper the flasks and allow to react for 5 minutes. Prepare the working sulfite-absorbing reagent solution (see below) at the same time iodine solution is added to the flasks. By means of a burette (50-ml burette) containing standardized 0.01 N thiosulfate, titrate each flask in turn to a pale yellow. Then add 5 ml of starch solution to the flask and shake thoroughly. Continue the titration until the blue color disappears. Store the standardized sulfite-TCM solution in a glass-stoppered bottle or flask.

3.11 Working Sulfite-TCM Solution

Pipette accurately 2 ml of the standard solution into a 100-ml volumetric flask and bring to mark with 0.04 M absorbing reagent. Calculate the concentration of sulfur dioxide in the working solution from the data in Section 3.10 as follows:

$$\mu g\ SO_2/ml = \frac{(A-B)\ (N)\ (32{,}000)}{25} \times 0.02$$

where A and B are averages of the three replicate titrations, and

A = volume thiosulfate for blank, ml,
B = volume thiosulfate for sample, ml,
N = normality of thiosulfate titrant,
32,000 = milliequivalent weight of SO_2, µg, and
0.02 = dilution factor.

This solution is stable for 30 days if kept at 5°C (refrigerator). If not kept at 5°C, prepare fresh daily.

3.12 Pararosaniline Stock Solution[a]

1. Place 100 ml each of 1-butanol and 1 N HCl in a large separatory funnel (250 ml) and allow to equilibrate. Note: Certain batches of 1-butanol contain oxidants that create an SO_2 demand. Before using, check by placing 20 ml of 1-butanol with 5 ml of 20 percent potassium iodide (KI) in a 50-ml separatory funnel and shake thoroughly. If a yellow color appears in the alcohol phases, redistill the 1-butanol from silver oxide, and collect the middle fraction, or purchase a new supply of 1-butanol.

2. Weigh 100 mg of pararosaniline hydrochloride (PRA) in a small beaker. Add 50 ml of the equilibrated acid (drain the acid from the bottom of the separatory funnel) to the beaker and let stand for several minutes.

3. To a 125-ml separatory funnel, add 50 ml of the equilibrated 1-butanol (draw the 1-butanol from the top of the separatory funnel). Transfer the acid solution containing the dye to the funnel, and extract. The violet impurity will transfer to the organic phase.

4. Transfer the lower (aqueous phase) into another separatory funnel and add 20 ml of 1-butanol; extract again. Repeat the extraction procedure with three more 10-ml portions of 1-butanol. This procedure usually removes almost all of the violet impurity that contributes to the blank (the extracted phase will still be red in color).

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[a] Some manufacturers offer high purity pararosaniline dye, which can be used in lieu of this purification step for the preparation of the standard curve as described in Section 5.3, provided the slope and intercept are within specifications.

5. After the final extraction, filter the acid phase through a cotton plug into a 50-ml volumetric flask and bring to volume with 1 N HCl. This stock reagent will be yellowish red.

3.13 Assay of Pararosaniline Stock Solution

1. Prepare a buffer stock solution with a pH of 4.69 by dissolving 13.61 g of sodium acetate trihydrate in distilled water in a 100-ml volumetric flask. Add 5.7 ml of glacial acetic acid and dilute to volume with water.

2. Take 1 ml of purified pararosaniline stock solution (3.12) and dilute to the mark in a 100-ml volumetric flask with distilled water.

3. Transfer a 5-ml aliquot to a 50-ml volumetric flask. Add 5 ml of 1 M acetate-acetic acid buffer solution from step 1 above and dilute the mixture to the mark with distilled water. Let the mixture stand for 1 hour.

4. Measure the absorbance at 540 nm with a spectrophotometer. Compute the percent of nominal concentration of PRA,

$$\%\ \text{PRA} = \frac{A \times K}{W}$$

where

A = measured absorbance of the final mixture, absorbance units

W = weight in grams of the dye in 50 ml of pararosaniline stock solution. For example, 100 mg of dye was used to prepare 50 ml of solution in the purification procedure, W = 0.100 g. If 0.5 g of dye is used to make 250 ml of pararosaniline stock solution, W is still 0.100 g.

K = conversion factor (molar absorptivity) whose value is dependent on the quality of the spectrophotometer and associated equipment.[7] For 1-cm cells and a spectral band width (half intensity spectral band width) of less than 11 nm, K = 21.3. **NOTE:** If the above specifications are not met, the dye analysis will be incorrect.

5. Perform this assay after each preparation or purchase of a new lot of PRA.

6. Record results of assay in calibration log book.

3.14 Pararosaniline Reagent

To a 250-ml volumetric flask, add 20 ml stock pararosaniline solution. Add an additional 0.2 ml stock solution for each percent the stock assays below 100 percent. Then add 25 ml of 3 M phosphoric acid and dilute to volume with distilled water. This reagent, stored in a glass-stoppered bottle, is stable for at least 9 months.

Table 3.1 ACTIVITY MATRIX FOR PREPARATION OF REAGENTS

Reagent	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Distilled water Reagent water preparation	Permanganate color (blue) does not appear after 1 hour at room temperature	Perform this test only if the slope and intercept of the standard curve fail to meet the specified criteria (Sec. 5.3)	Repeat the purity test until the slope and intercept meet the required limits
Reagent water storage	Inert containers with vent guard	Visual check, cover and vent guard in place and functioning	Replace vent guard. drying materials
Absorbing reagent pH test	$3.0 < pH < 5.0$ (optimal $pH = 4.0$)	Check new reagent before use with pH meter	Dispose, check chemicals, prepare new lot
Storage	1) Less than 6 mos. since preparation 2) Absence of precipitate	Visual check for preparation date and precipitate	Dispose
Sulfamic acid	0.6%, keep in glass stoppered flask	Gravimetric preparation, fresh daily	Prepare fresh reagent
Formaldehyde	0.2%, keep in stoppered container	Volumetric preparation, fresh daily	As above
Starch indicator solution	2 g/liter clear solution	Gravimetric preparation	As above
Stock iodine solution	0.1 N, keep in glass-stoppered dark bottle	As above	As above
Working iodine solution	0.01 N, keep in glass-stoppered dark flask	Volumetric preparation, prepared fresh daily	As above
Stock sodium thiosulfate	0.1 N, keep in glass-stoppered flask	Standardized monthly against potassium iodate standard	As above
Sodium thiosulfate titrant	0.01 N, keep in glass-stoppered flask	Volumetric preparation, prepared daily	As above
Standard sulfite-TCM solution	320 to 400 μg/ml SO_2 equivalent	Standardize by titration	As above
Working sulfite-TCM solution	Accurate preparation from standardized stock solution	Volumetric preparation, prepared day of use (or stored < 30 days @ 5°C)	As above
Stock pararosaniline solution	0.2% concentration	Assay at preparation	As above
Assay of pararosaniline stock solution		Perform after each preparation or purchase of new lot of PRA	
Pararosaniline reagent	100% assay	Assay at preparation and monthly beginning at 9 months	As above

4.0 SAMPLING PROCEDURE

4.1 Selection of Sampling Train

A midget impinger sampling train, Figure 4.1, is specified for short-term sampling, 3 hours or less. Flow control for this sampling train is maintained by means of a critical flow orifice or a needle valve. The flow rate is normally monitored by means of a rotameter.

The sampling train commonly employed for 24-hour sampling periods consists of polypropylene bubbler tubes and uses critical orifices for flow control. An example of this sampling train configuration is presented in Figure 4.2.

4.2 Apparatus Assembly

4.2.1 Absorber Assembly -

1. For short-term sampling, 30 minutes and 1 hour, use 30-ml all glass midget impingers[a] as specified in the Reference Method, reprinted in Section 2.1.10.
2. For 24-hour samples, select absorber consisting of:
    - a. Polypropylene container 164-mm depth by 32-mm diameter.
    - b. Two-port polypropylene tube closure (these are commercially available).
    - c. Glass impinger tube, 6-inch length by 6-mm diameter with outlet orifice diameter to fit No. 79 jeweler drill. (A No. 78 drill is too large.) Orifice size is between 0.368 and 0.406 mm.

4.2.2 Cleaning Absorber - Before each use:

1. Wash in hot water.
2. Wash in acid bath 1 hour.
    - a. 1 part nitric acid.
    - b. 2 parts hydrochloric acid.
    - c. 4 parts distilled water.
3. Rinse with distilled water.
4. Air dry (drain).

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[a] Nozzle of impinger calibrated to deliver 0.09 to 0.11 cfm (2.5 to 3.1ℓ/m) at 12 in. H_2O vacuum. The orifice of the impinger nozzle is approximately 2 mm.

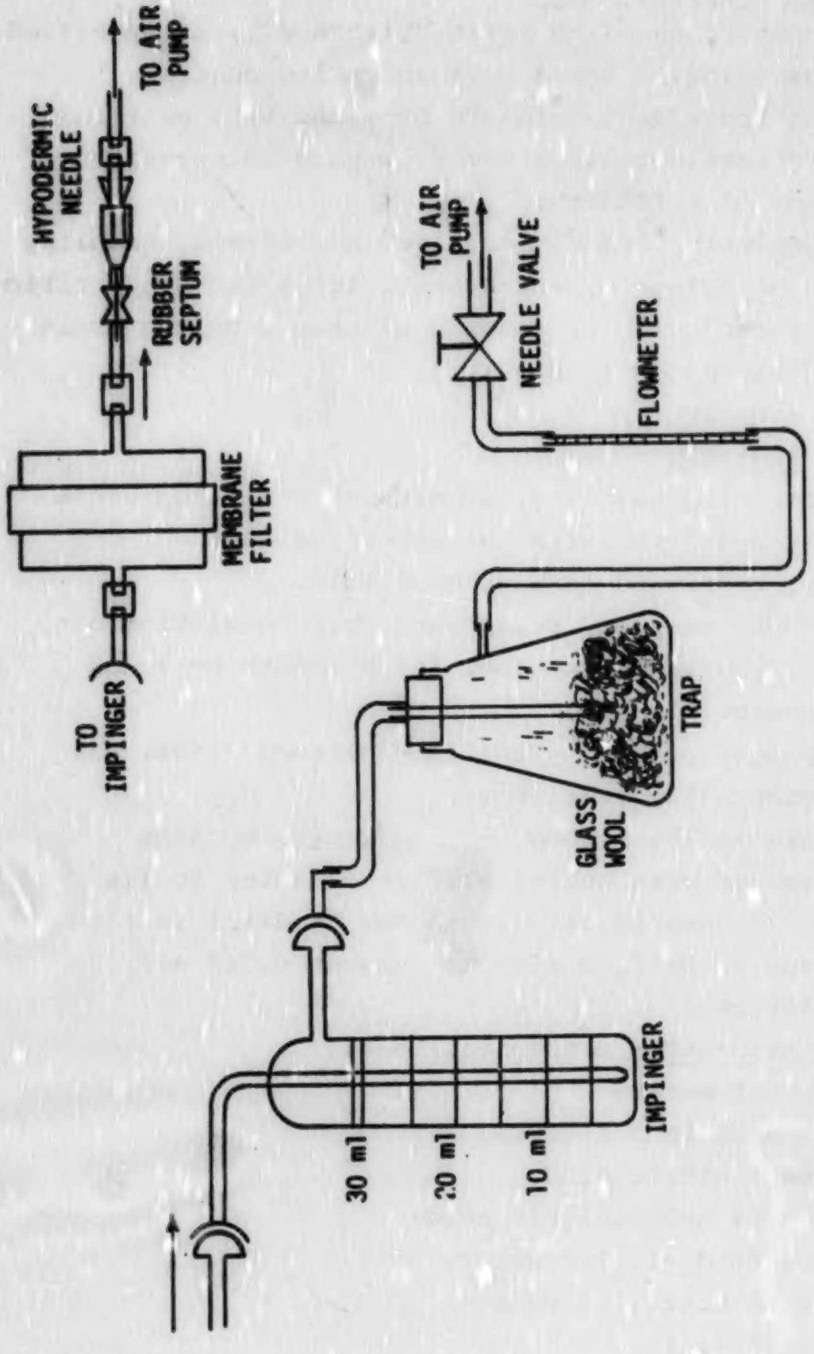

TO AIR PUMP
HYPODERMIC NEEDLE
RUBBER SEPTUM
MEMBRANE FILTER
TO IMPINGER
TO AIR PUMP
NEEDLE VALVE
FLOWMETER
TRAP
GLASS WOOL
30 ml
20 ml
10 ml
IMPINGER

Figure 4.1 Midget impinger sampling train

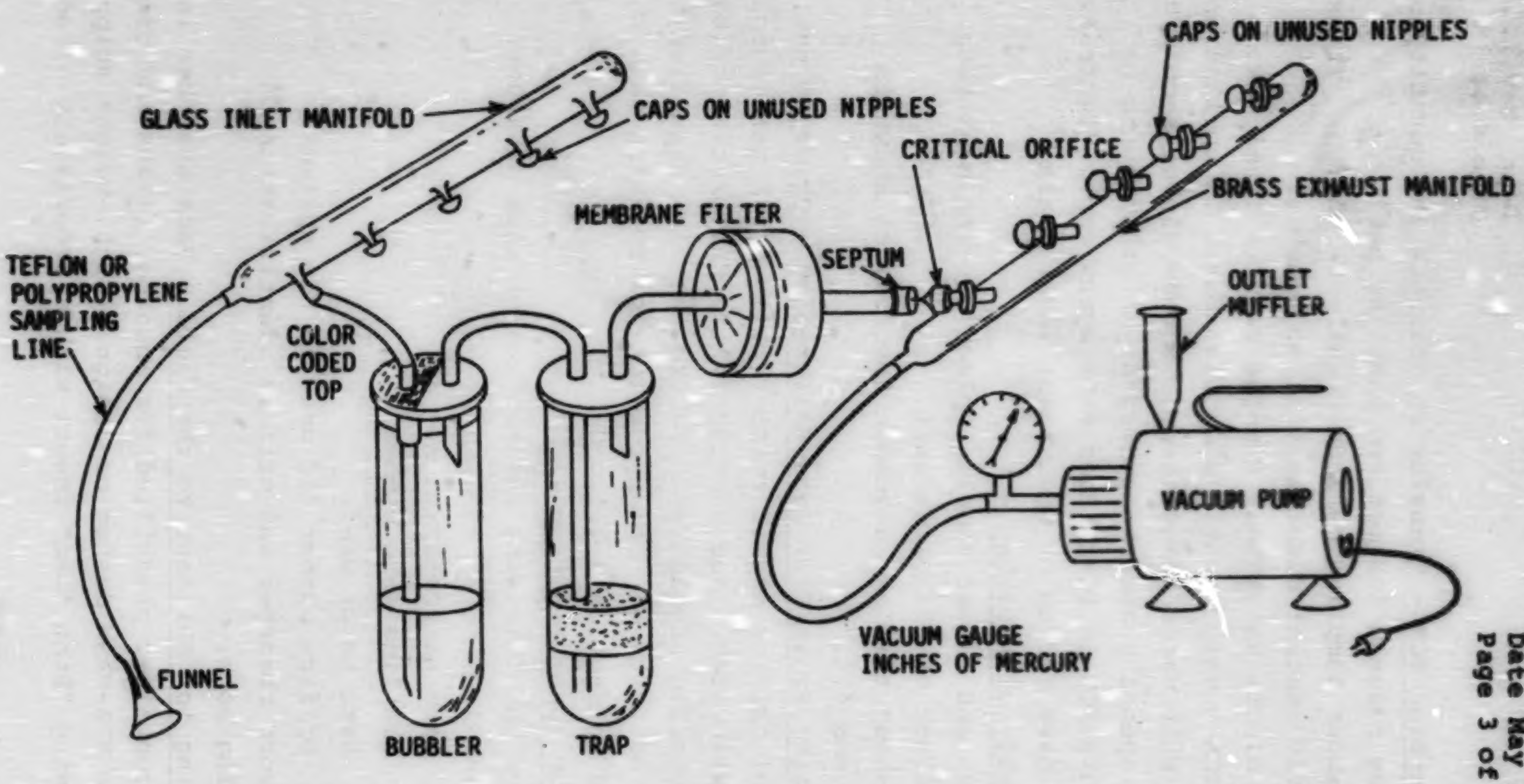

GLASS INLET MANIFOLD
CAPS ON UNUSED NIPPLES
CAPS ON UNUSED NIPPLES
CRITICAL ORIFICE
BRASS EXHAUST MANIFOLD
MEMBRANE FILTER
SEPTUM
OUTLET MUFFLER
TEFLON OR POLYPROPYLENE SAMPLING LINE
COLOR CODED TOP
VACUUM PUMP
VACUUM GAUGE INCHES OF MERCURY
FUNNEL
BUBBLER
TRAP

Figure 4.2 Bubbler sampling train

4.2.3 Dispensing TCM - Transfer to absorber the quantity of TCM absorbing reagent for desired sampling period:

1. 24-hour sampling periods require 50 ml TCM. Transfer with automatic reagent dispensers is acceptable.

2. 30-minute and 1-hour samples require 10 ml TCM. (WARNING: TCM highly caustic and poisonous. If spilled on skin, flush with water immediately. Rubber gloves should be worn when dispensing absorbing reagent.) If shipping is involved, ship TCM in test tubes with screw caps, Figure 4.3. Caps should have Teflon or equivalent inner seals.

4.2.4 Assembly of Absorber -

1. 1/2- and 1-hour samples, place cap-impinger tube element in flask of all glass unit.

2. 24-hour samples, assemble absorber as shown in Figures 4.3 and 4.4.

4.2.5 Sampling Train Flow Rate Calibration - Prior to use, each critical orifice and/or rotameter, in short term sampling train, is calibrated. See Section 2.1.2, Calibration of Equipment, for detailed procedures. Record the flow data from the calibration activity on the appropriate calibration data sheet as presented in Section 2.1.12.

4.2.6 Sample Identification -

1. Mark the following information on each absorber. Use a stick-on label.

   a. Date of preparation.
   b. Sampling site number.
   c. Date to be used.
   d. Orifice number (if used in the system).

2. Record absorber and orifice identification on laboratory log book.

4.2.7 Packing for Shipment to the Field - Pack absorber in wood block container predrilled for absorber flasks and critical orifices per EPA-NASN design or design a light-weight shipping container using "Etha foam" insert with appropriately sized

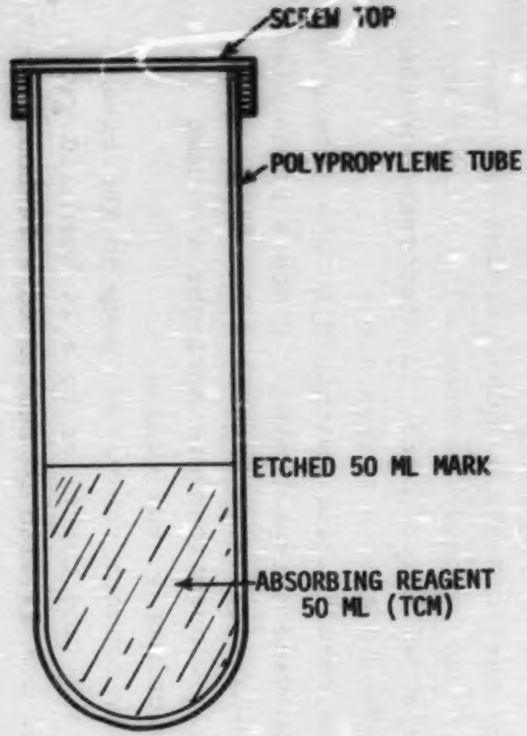

SCREW TOP
POLYPROPYLENE TUBE
ETCHED 50 ML MARK
ABSORBING REAGENT
50 ML (TCM)

Figure 4.3 Screw top bubbler for shipment

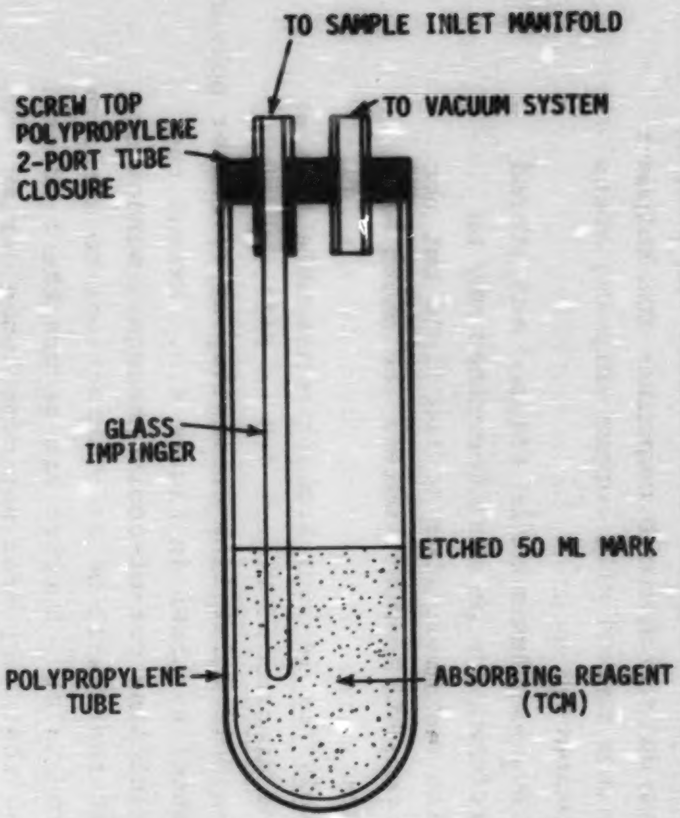

TO SAMPLE INLET MANIFOLD
TO VACUUM SYSTEM
SCREW TOP
POLYPROPYLENE
2-PORT TUBE
CLOSURE
GLASS
IMPINGER
ETCHED 50 ML MARK
POLYPROPYLENE
TUBE
ABSORBING REAGENT
(TCM)

Figure 4.4. Absorber for 24-hour sampling

holes to support the bubbler tubes. This insert can then be inserted into a heavy-duty fiberboard container for shipment to the field. The latter method will reduce shipping costs by a factor of approximately 5.

CAUTION: Collected SO_2 samples must be shipped and stored at 5°C or less; therefore, the process described may be applicable to shipping absorber tubes to the field but not returning the collected SO_2 samples, unless ice packs are used with the container.

4.2.8 Sampler Assembly for 24-hour Bubbler - Assemble sampler elements shown in the diagram of Figure 4.5 as follows:

1. Attach the sample inlet line consisting of Teflon or polypropylene tubing and funnel as shown in Figure 4.5. Extend the funnel and sample inlet line out-of-doors through a window or other opening so that there will be no obstructions to air flow in the vicinity of the inlet. Support and secure the tube and funnel in position so that they will not come loose during sampling. The funnel should be positioned to hang down so that rain will not be drawn into the sampler. If the sampler is to be used outdoors, a temperature controlled shelter must be used. These shelters and sampling trains are commercially available, or can be built incorporating a small refrigerator or a cooler with ice packs. A photograph of the absorber installation appears in Figure 4.6.

2. Connect the metal exhaust manifold to the intake of the vacuum pump with vacuum type tubing. Place a pinch clamp on this section of tubing as shown in Figure 4.5, but do not tighten. Be sure all connections are airtight and that there are no constrictions in the tubing.

[illegible] Electrically connect the vacuum pump to the timer switch (see Figure 4.5) and the timer to a 24-hour 110 VAC outlet. The sampler box is also connected to a 24-hour outlet.

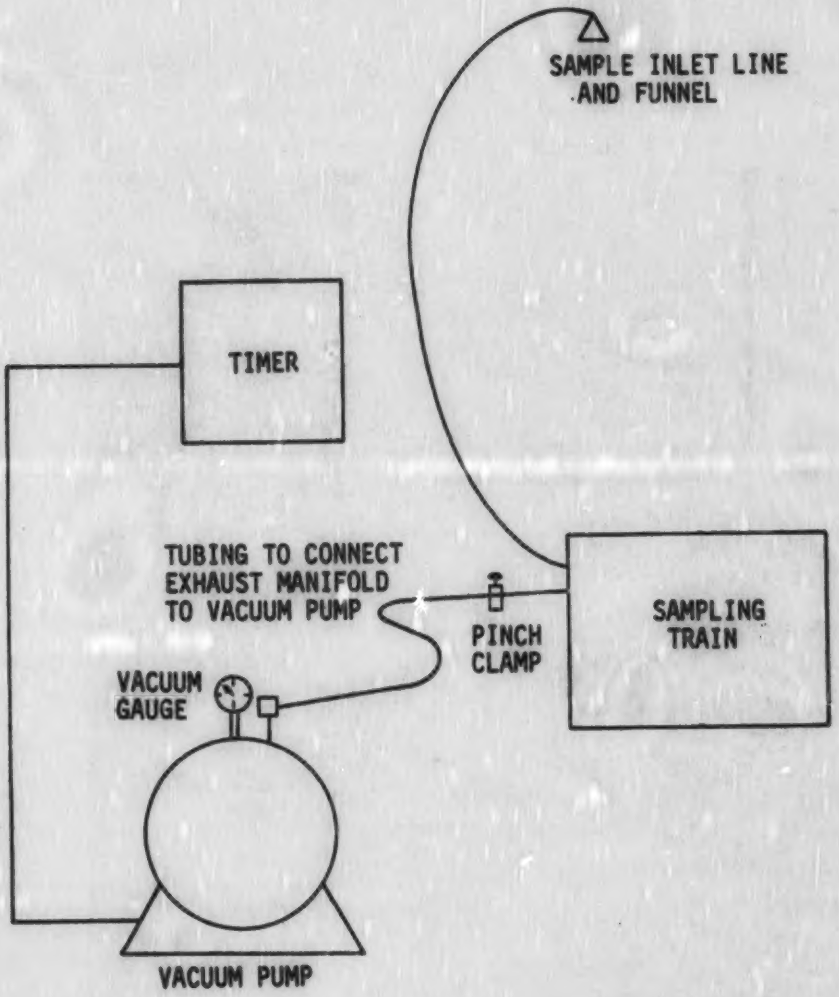

SAMPLE INLET LINE AND FUNNEL
TIMER
TUBING TO CONNECT EXHAUST MANIFOLD TO VACUUM PUMP
PINCH CLAMP
SAMPLING TRAIN
VACUUM GAUGE
VACUUM PUMP

Figure 4.5 Diagram of SO_2 sampling apparatus

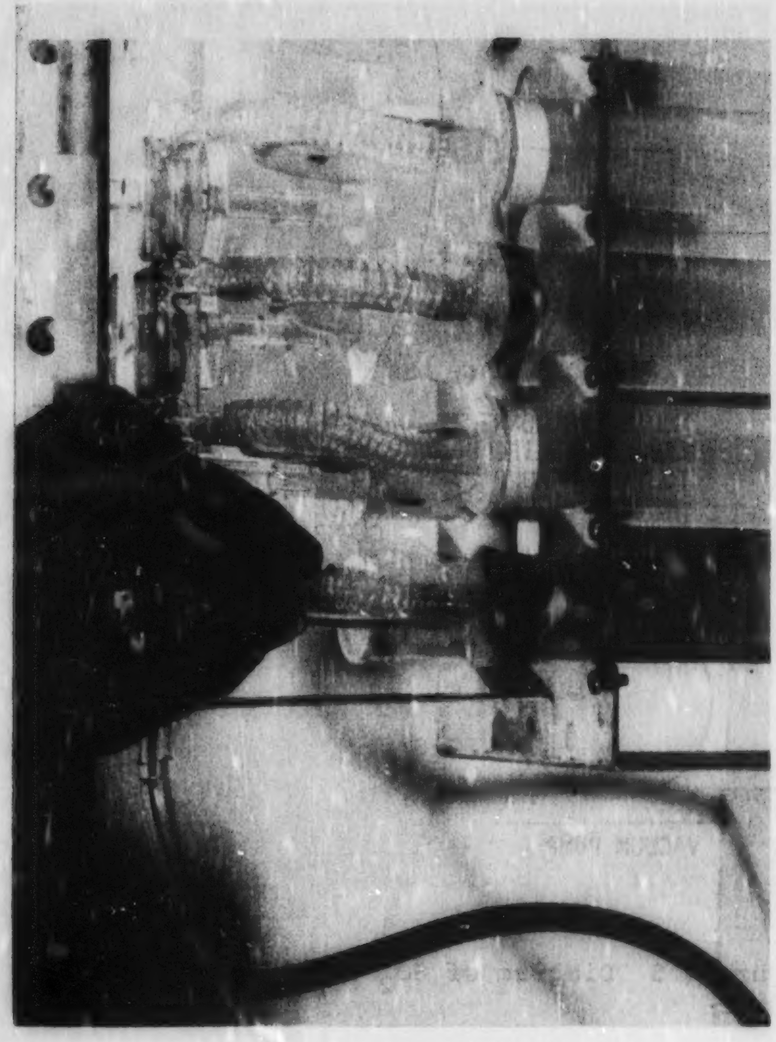


Figure 4.6. Close-up view of absorber installation.

4.2.9 Installation of Absorber and Critical Orifice - Absorber and sampling apparatus tubing should be color coded to facilitate proper positioning of absorbers and correct tubing connections (Figure 4.6). Accordion type tubing is used to connect the glass intake manifold to the impinger tube of the absorber. Smooth tubing is used for all other connections.

A stepwise installation procedure for the 24-hour bubbler is presented below.

1. Remove the dummy absorber (or used absorber) from the sampling train by gently but firmly pulling the inlet and outlet tubing from the absorber.
2. Exchange the reagent-filled absorber for the dummy absorber.
3. Transfer the caps from the reagent-filled absorber to the dummy absorber. NOTE: Always install the tube cap or tubing on the impinger tube (color-coded side of absorber) first and remove them last, otherwise some of the absorbing reagent/sample may be forced out the impinger opening and be lost.
4. Gently but firmly insert the accordion inlet tube (from the glass manifold) onto the color-coded tube (impinger tube) of the absorber lid. Make certain the fit is airtight.
5. Gently but firmly insert the smooth tubing (from the trap) to the unpainted outlet tube of the absorber.
6. Remove the critical orifice (calibrated hypodermic needle) from the shipping container. NOTE: It is important that the needle be used and returned with the same absorber.
7. Insert the new needle into the center of the rubber septum attached to the membrane filter unit as shown in Figure 4.7. The needle must be put in straight. If the needle is accidently bent during installation, it should be destroyed and discarded and another calibrated absorber and needle used.
8. Slide the base of the needle onto the metal exhaust manifold as shown in Figure 4.8. Manipulate the needle and/or rotate the trap, if necessary, to obtain a tight connection.

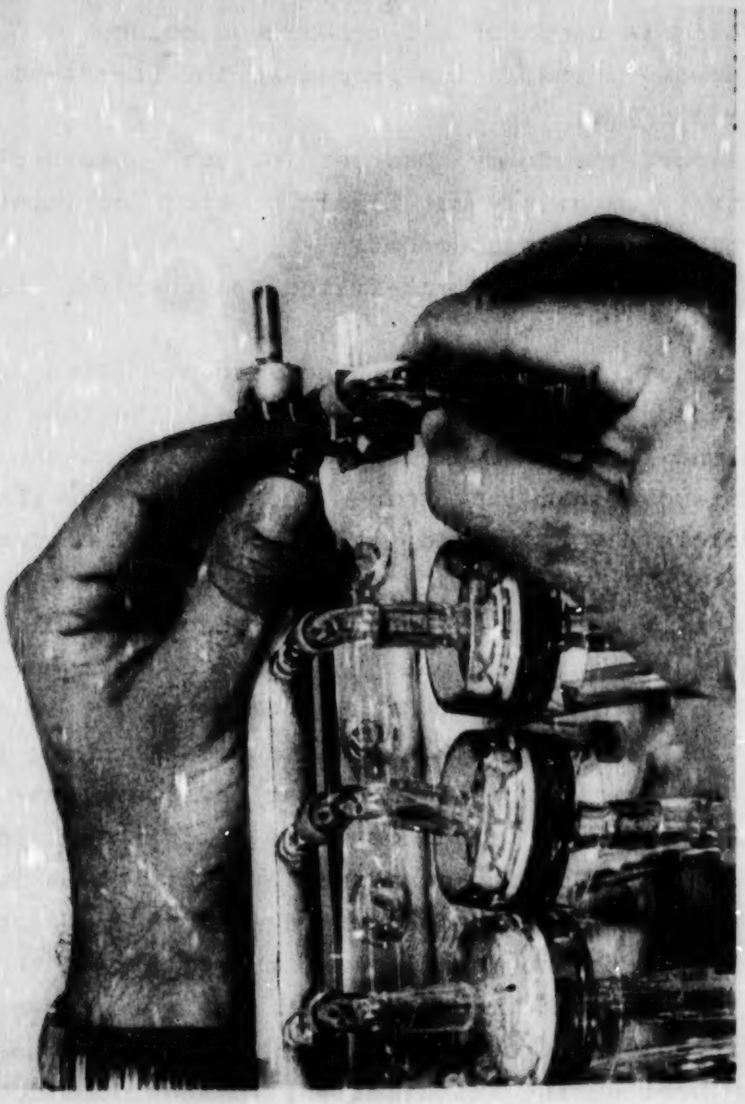


Figure 4.7. Installation of critical orifice in the sampling train

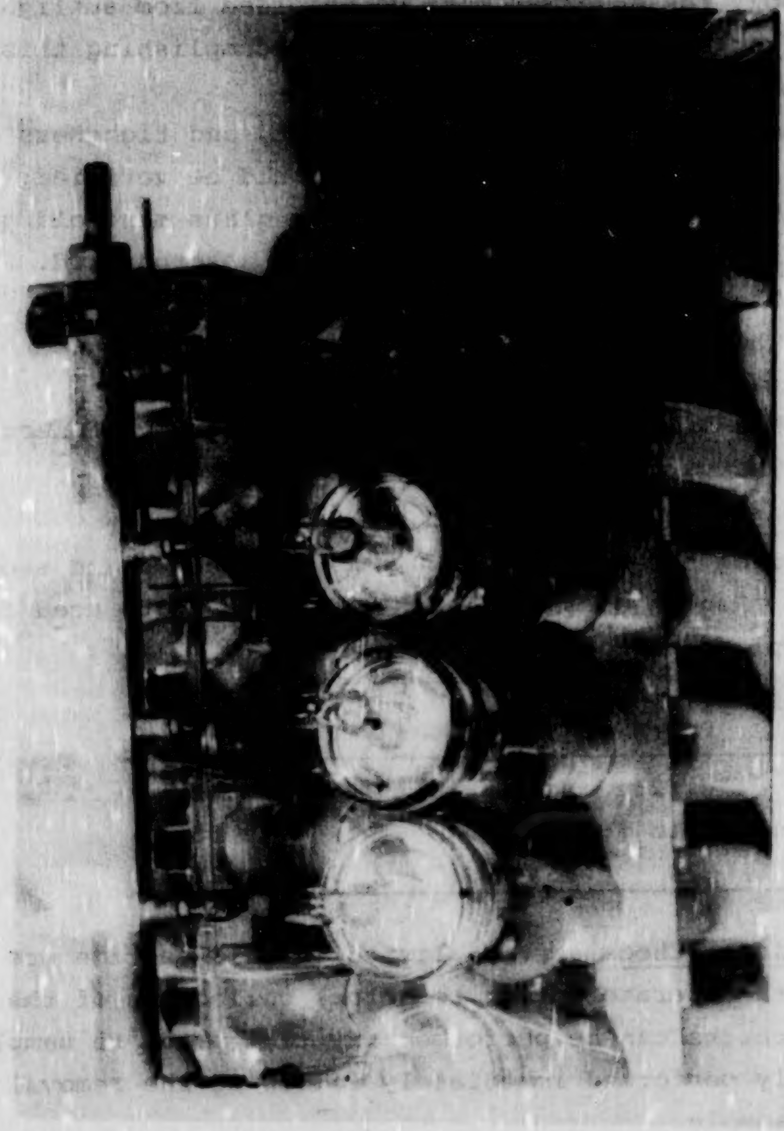


Figure 4.8. Connecting critical orifice to exhaust manifold.

9. Leak check the total system by activating the vacuum pump, capping off the plastic sample inlet nipple, and visually checking for bubbling. Should a leak be detected check all fittings for leaks (see Section 4.3.1).

10. In situations where the sampling train is not housed in a closed box, the absorber must be shielded from sunlight during and after sampling. One means of accomplishing this is to wrap the absorber in aluminum foil.

11. Recheck the arrangement, alignment, and tightness of all connections. The following points should be verified:

   a. The accordian tubing from the glass manifold goes to the color-coded side of the absorber lid.
   b. The needle is not bent or obstructed.
   c. The needle forms a tight fit at the exhaust manifold.
   d. The inlet filter and tubing is tightly connected.
   e. The vacuum pump connections are tight.

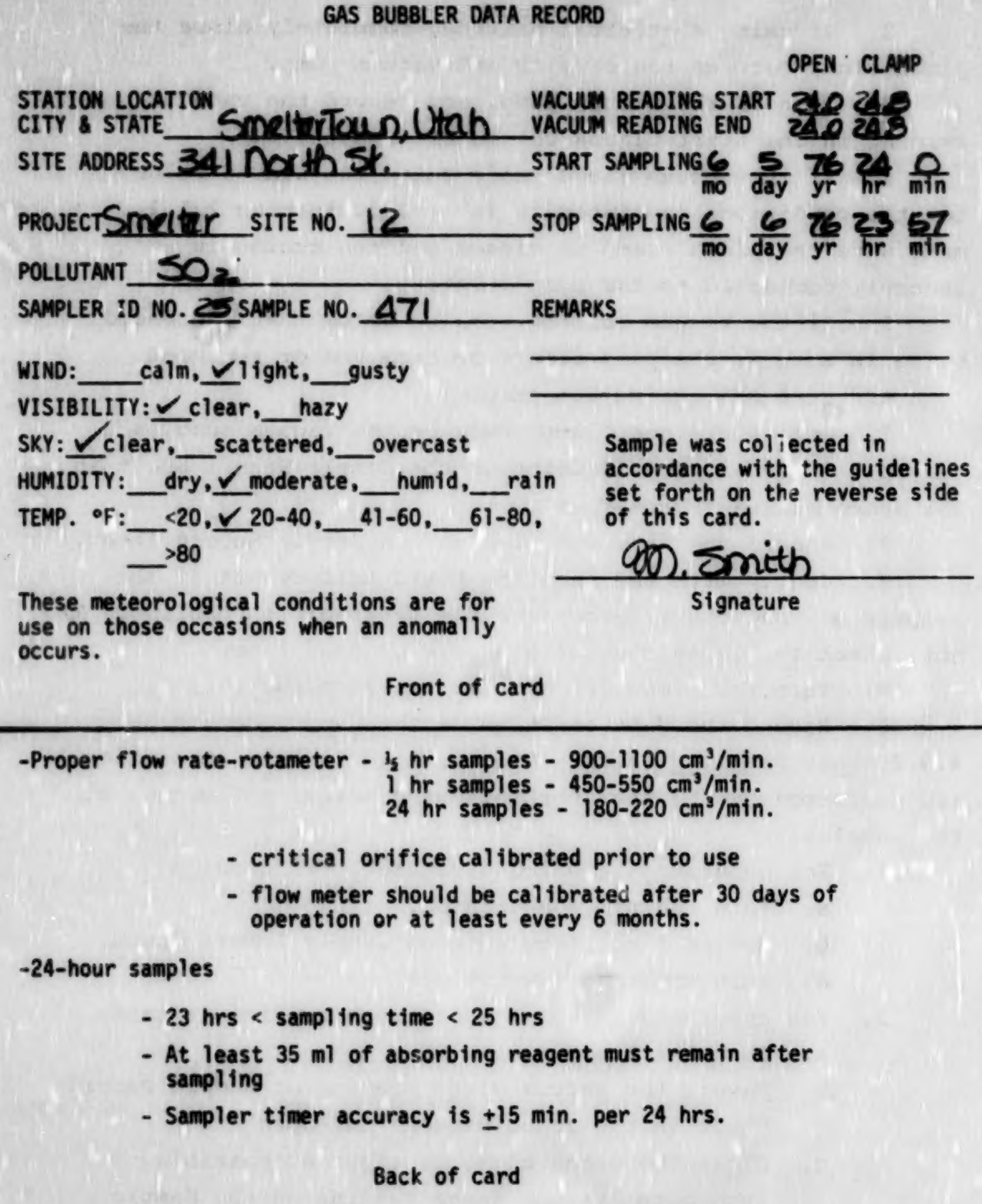
4.3 Sample Collection

Operation of the sampling apparatus is started and stopped by the timer. Ranges of sample air flow rates to be used for different sampling periods are:

Sampling period hours	Flow rate range cc/min
1/2	900 to 1100
1 to 3	450 to 550
24	180 to 220

4.3.1 Operational Check - Steps listed in this section are performed by the operator prior to actual collection of the sample. These checks can be performed any time prior to sampling and are normally performed immediately following the removal of the prior sample.

1. If a rotameter equipped with needle valve is used, adjust the system flow rate to the prescribed value on the Sample Record Sheet indicated on back of card, Figure 4.9.



GAS BUBBLER DATA RECORD

STATION LOCATION
CITY & STATE Smelter Town, Utah
SITE ADDRESS 341 North St.
PROJECT Smelter SITE NO. 12
POLLUTANT SO_2
SAMPLER ID NO. 25 SAMPLE NO. 471

	OPEN	CLAMP
VACUUM READING START	24.0	24.8
VACUUM READING END	24.0	24.8

	mo	day	yr	hr	min
START SAMPLING	6	5	76	24	0
STOP SAMPLING	6	6	76	23	57

REMARKS

WIND: ___calm, ✓ light, ___gusty
VISIBILITY: ✓ clear, ___hazy
SKY: ✓ clear, ___scattered, ___overcast
HUMIDITY: ___dry, ✓ moderate, ___humid, ___rain
TEMP. °F: ___<20, ✓ 20-40, ___41-60, ___61-80, ___>80

These meteorological conditions are for use on those occasions when an anomally occurs.

Sample was collected in accordance with the guidelines set forth on the reverse side of this card.

M. Smith
Signature

Front of card

-Proper flow rate-rotameter - ½ hr samples - 900-1100 cm^3/min.
1 hr samples - 450-550 cm^3/min.
24 hr samples - 180-220 cm^3/min.

- critical orifice calibrated prior to use
- flow meter should be calibrated after 30 days of operation or at least every 6 months.

-24-hour samples

- 23 hrs < sampling time < 25 hrs
- At least 35 ml of absorbing reagent must remain after sampling
- Sampler timer accuracy is ±15 min. per 24 hrs.

Back of card

Figure 4.9. Sample record sheet

2. If using a critical orifice, completely close the pinch clamp between the sampler and vacuum pump.

3. Turn ON the vacuum pump, and record the vacuum reading in the Start-Clamps column of Figure 4.9.

4. If the vacuum gauge reads below the stated value for the conditions as listed in Table 2.3, Section 2.1.2, make sure the pinch clamp is closed and the tubing is securely connected to the pump inlet.

5. If the vacuum reading remains below the referenced value in step 4, the pump should be repaired or replaced.

6. Turn OFF the vacuum pump.

7. Open the pinch clamp, turn on the vacuum pump, and record the vacuum gauge reading on the Sample Record Sheet in the space marked "Start-Open".

8. Record the date and time on the Sample Record Sheet.

9. Gently lift the sampling train halfway out of the sampler box and check to be sure the absorber is bubbling. If not, check for loose connections or a plugged line.

10. Turn the timer OFF and set for sampling period.

11. Record the date and time on the Sample Record Sheet.

4.3.2 Post Sampling Check - Steps listed in this subsection are performed by the operator following actual collection of the sample.

1. For apparatus in which a rotameter is used:
   a. Turn the timer switch ON.
   b. Record final flow rate on Sample Record Sheet.
   c. Turn the timer switch OFF.
2. For apparatus in which a critical orifice is used:
   a. Turn the timer switch ON.
   b. Record the vacuum gauge reading on Sample Record Sheet in the space marked "End-Open".
   c. Close the pinch clamp as tight as possible. Record the vacuum gauge reading on the Sample Record Sheet in the space marked "End-Clamp".
   d. Turn the timer switch OFF.

3. Check the condition of the membrane filter. Replace the filter if it is discolored or cracked.

4. If the ambient temperature is lower than the thermostat setting in the sampling box, check to assure that the thermostat and heater are functioning.

5. Record an average temperature of the absorbing solution for the sampling period. For 1/2- and 1-hour samples, the temperature immediately before or after, or at any time during the sampling period is adequate. For 24-hour samples the use of a recording thermometer is recommended within the sampling train shelter. Record the mean of readings from the recording thermometer kept in the sampler box. A minimum-maximum thermometer has also been used for this purpose but it is only an indication of the degree of temperature control within the sampler box. If the minimum-maximum thermometer readings are unavailable, record the normal thermostatic setting of the sampler box or the ambient temperature for the sampling period, whichever is greater.

NOTE: Unfortunately, the SO_2-TCM complex is thermally unstable. The SO_2 concentration measured in an air sample is very dependent upon the temperature history of the TCM absorbing solution during sampling and after sample collection. SO_2 is lost from collected samples at rates that are highly dependent upon temperature and independent of concentration as long as EDTA is used as called for, as the following table[8] shows:

Temperature		Rate of SO_2 loss
°C	°F	%/day
20	68	0.9
30	86	5.0
40	104	25.0
50	122	73.6

Separate experiments show that if the temperature of the absorbing solution is 40°C (104°F) during sampling, using a 24-hour sampling period, only about 80 percent of the theoretical amount of SO_2 is recovered.[8]

These studies show that careful temperature control is required during sample collection and storage. We recommend that agencies making SO_2 measurements using the pararosaniline method carry out the sampling in such a manner that the TCM absorbing reagent is maintained at 25°C or less and collected samples after removal from the sampling train are maintained at 5°C or less in a refrigerator until analysis.

6. Record the barometric pressure for the sampling period as obtained with a calibrated instrument or from the local weather bureau.

7. Check the absorber for any reagent loss due to evaporation. Record on the Sample Record Sheet an estimate of the quantity of absorbing reagent or state under "Remarks" that there was no evaporation.

4.3.3 Sampling Time Period - Record sample time period on Sample Record Sheet. For 24-hour samples, void sample if sampling period is less than 23 hours or more than 25 hours.

4.4.4 Sample Handling -

1. First remove the accordian tubing (color coded) from the absorber impinger tube.

2. For 24-hour samples, estimate the volume of absorber reagent remaining in the absorber flask after sampling and record on Sample Record Sheet. To assist in this estimation mark the tubes at the 35- and 50-ml levels with a marking pen. If less than 35-ml, void the sample, and so state on the Sample Record Sheet. Mark TCM level in absorber with tape or some other easily removable indicator.

3. For 24-hour samples having 50-ml of sampling reagent remaining after sampling, state under "Remarks" on the Sample Record Sheet that no evaporation occurred in sampling.

4. If there is evidence of malfunction, e.g., absorbing reagent forced out of absorber into the vacuum system, record this malfunction on the Sample Record Sheet. Normally the sample is void under this circumstance.

5. Place tube cap first on impinger tube (color coded) of absorber, then on suction tube. Press caps firmly in place and place absorber in shipping block. (NOTE: For 1/2- and 1-hour samples collected in glass impingers, shake the absorber thoroughly, then quantitatively transfer all the exposed reagent to a test tube or small bottle with a Teflon-lined, threaded cap. Place the sample in the shipping block.)

6. If a critical orifice is used, disconnect the base end of the hypodermic needle from the metal exhaust manifold and remove the needle from the rubber stopper. Place the needle in a protective shield (these are commercially available with most needles) and then place it in the shipping block with the exposed absorber with which it was used.

7. Record on the Sample Record Sheet any pertinent observations relative to sources, weather conditions, etc., that might affect the SO_2 level.

8. Complete the Sample Record Sheet in duplicate; return the original copy with the absorber in the shipping block; file the duplicate in the site log book.

9. Place the sample in a cooler and deliver the sample to the laboratory, or pack the absorber (and critical orifice) in an insulated shipping container with ice packs and ship it promptly to the receiving laboratory. Should delays between sampling and shipment to the laboratory occur, the samples should be stored at 5°C in a refrigerator until shipment.

Table 4.1 ACTIVITY MATRIX FOR SAMPLING PROCEDURE

Activity	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Absorber selection	1) Short term, less than 3 hours, use 30-ml all-glass midget impinger 2) 24-hr samples, polypropylene tube and closure with glass impinger tube 3) Orifice ID to larger than #79 drill and smaller than #78 drill	Visually check each sample	Select proper absorber
Cleaning absorber	Clean absorber	Water and acid bath prior to each use	Perform cleaning
Dispensing TCM	1) Short-term periods require 10 ml TCM 2) 24-hr sampling periods require 50 ml TCM	Visually check each sample	Correct discrepancy
Assembly of absorber	As diagramed	As above	Reassemble
Rotameter vs. wet test meter	All data points within ±2% of best-fit curve	Prior to each use re-calibrate as per Section 2.1.4	Rerun deviate points/recalibrate
Critical orifice vs. soap bubble meter, rotameter or mass flow meter	As per Section 2.1.5	As above but recalibrate as per Section 2.1.5	As above
Sample ID (stick-on label)	All required information	Visual check	Complete label
Packing for shipment	Ship in insulated cool pack container	As above	Repack as appropriate
Sampler assembly 24-hr bubbler	NASN specifications as per Figures 4.5, 4.6, and 4.7	As above	Reassemble if necessary
Installation of absorber and critical orifice	See Section 4.2.8	As above	Reassemble and/or select proper orifice
Sample collection	Air flow rates as specified in Section 4.3	As above	Adjust flow rate
Sample storage prior to analyzing	Store at 5°C	As above	Correct discrepancy

5.0 ANALYSIS OF SAMPLES

A summary of the quality control functions for analysis of samples is contained in Table 5.1 at the end of this section.

5.1 Verification of Field Record Sheet

1. Examine Sample Record Sheet and invalidate sample if vital information is missing and cannot be obtained from field personnel.

2. Log sample into sample receiving log book.

3. Verify the absorber volume marked on absorber and recorded on the Sample Record Sheet versus the sample volume as received. If the sample volume is lower than marked on the absorber, leakage occurred. Void the sample. For valid samples that have less than 50 ml absorbing reagent add distilled water to bring to 50-ml and continue with analysis.

5.2 Determination of Sampling Rates

5.2.1 Flow Rate Check for Systems with Critical Orifice - (For samples taken with critical orifice flow rate control.)

1. Repeat the flow rate measurement as described in either the soap bubble meter, mass flow meter or rotameter technique.

2. Determine the average flow rate ($\bar{Q}_f$) at standard conditions

$$\frac{Q_1 + Q_2}{2} = \bar{Q}_f \qquad \text{Equation 5-1}$$

where

Q_1 = initial flow rate at standard conditions (760 mm Hg and 25°C)

Q_2 = final flow rate at standard conditions (760 mm Hg and 25°C)

3. Compute the percent change between the initial flow rate and that after sampling (final rate) as follows,

$$\%\ \text{change} = \frac{{}^{a}|Q_1 - Q_2|100}{Q_1} = \qquad \text{Equation 5-2}$$

---

[a] $|Q_1 - Q_2|$ means the numerical or absolute value of the difference, i.e., a positive number or zero.

If % change is ≥ 10, invalidate the sample.

If % change is < 10, use the average flow rate to determine the SO_2 concentration in the sample.

5.2.2 Flow Rate Check for Flow Systems with Rotameters -

1. Repeat the flow rate measurement as described in Section 2.1.4 and determine the final flow rate corrected to standard conditions.

2. From the initial and final rotameter readings taken from the rotameter calibration curve determine the average final flow rate at standard conditions using equation 5-1.

3. Compute the percent change in the flow rates from the initial Q_1 and the final rotameter reading Q_2, using equation 5-2.

5.3 Colorimetric Analyses - Calibration curve for spectrophotometer

The following stepwise procedure is presented for obtaining the calibration relationship of μg SO_2 versus absorbance.

1. Prepare six 25-ml volumetric flasks with 0, 0.5, 1, 2, 3, and 4 ml working sulfite-TCM, respectively.

2. Add TCM absorbing solution to each flask such that the approximate total volume is 10 ml.

3. Add 1 ml of 0.6 percent sulfamic acid. Allow to stand 10 minutes.

4. Add 2 ml of 0.2 percent formaldehyde.

5. Add 5 ml of pararosaniline solution and start timer.

6. Add distilled water to 25-ml mark.

7. Place in a water bath for at least 30 minutes but not longer than 60 minutes at 20 ± 0.2°C.

8. Allow at least 5 minutes for the spectrophotometer to warm up. Adjust the zero control to bring the meter needle to infinity absorbance on the scale. Standardize the light control by inserting a cell filled with distilled water into the sampler holder and adjusting the light control as required, until the meter reads zero absorbance. NOTE: The complete absorbance scale should be checked with a calibrated set of filters from the National Bureau of Standards any time a

control sample cannot be measured within ± 1.2 μg SO_2 of its known value (see Section 2.1.9). Measure a reagent blank and a control sample before each set of determinations.

9. Remove the samples from the water bath and immediately read, employing one centimeter optically matched cuvettes,[a] the absorbance at 548 nm for each solution and record on the laboratory data log, Figures 5.1A and 5.1B.

10. Plot absorbance versus the μg SO_2 contained in each 25-ml flask. To compute the μg SO_2 per flask, refer to Section 3.13 to determine the concentration of the working TCM sulfite solution.

11. Use regression analysis to determine slope and intercept of the calibration curve. Refer to Appendix J of Volume I of the Handbook[9] for discussion of linear regression analysis.

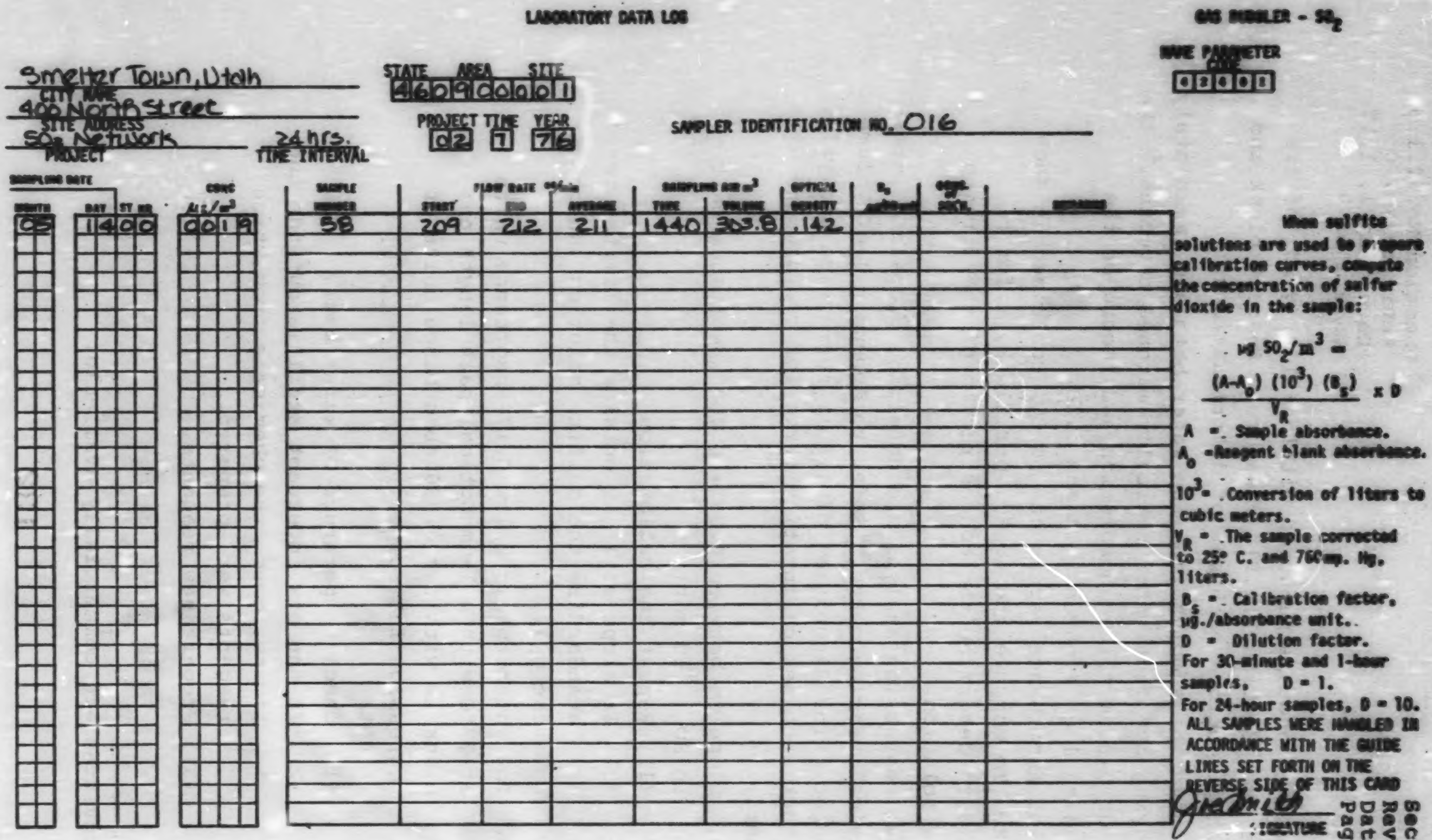
12. Check each new calibration curve for conformance with these criteria:

   a. Slope is between 0.03 ± 0.002 absorbance unit/μg SO_2. If not:
      (1) Repeat the calibration
      (2) If slope still is outside the limits, restandardize the sulfite solution and check the dye.

   b. The intercept is below 0.17 absorbance units at 22°C with a 1-cm optical path length and blank is within ± 0.030 absorbance units of regression intercept.
   It not:
      (1) Check temperature of water bath.
      (2) If the new intercept still is outside the limits, check the chemicals from which reagents were prepared.
      (3) Prepare new reagents.

---

[a] Matched cuvettes are commercially available.



LABORATORY DATA LOG

GAS BUBBLER - SO_2

[illegible] PARAMETER CODE 42401

Smelter Town, Utah
CITY NAME

400 North Street
SITE ADDRESS

SO_2 Network
PROJECT

24 hrs.
TIME INTERVAL

STATE 46 AREA 0900 SITE 001

PROJECT 02 TIME 1 YEAR 76

SAMPLER IDENTIFICATION NO. 016

SAMPLING DATE MONTH	DAY	ST HR	CONC µg/m³	SAMPLE NUMBER	FLOW RATE cc/min START	END	AVERAGE	SAMPLING AIR m³ TIME	VOLUME	OPTICAL DENSITY	B_s	CONC. OF SOLN.	REMARKS
05	14	00	0019	58	209	212	211	1440	303.8	.142			

When sulfite solutions are used to prepare calibration curves, compute the concentration of sulfur dioxide in the sample:

$$\mu g\ SO_2/m^3 = \frac{(A-A_o)\ (10^3)\ (B_s)}{V_R} \times D$$

A = Sample absorbance.
A_o = Reagent blank absorbance.
10^3 = Conversion of liters to cubic meters.
V_R = The sample corrected to 25° C. and 760mm. Hg. liters.
B_s = Calibration factor, µg./absorbance unit.
D = Dilution factor.
For 30-minute and 1-hour samples, D = 1.
For 24-hour samples, D = 10.

ALL SAMPLES WERE HANDLED IN ACCORDANCE WITH THE GUIDE LINES SET FORTH ON THE REVERSE SIDE OF THIS CARD

SIGNATURE

Figure 5.1A Laboratory Data Log (Front).

<table>
<tr><th>PREPARATION AND CALIBRATION</th><th>SAMPLE ANALYSIS</th><th>REAGENT PREPARATION</th></tr>
<tr>
<td>Reagent water must be double distilled and be stored in a borosilicate glass vessel, capped and with a vent guard.
The absorbing reagent must be prepared every 6 months, be clear and have 3.0 pH < 5.0.
24-hour samples were polypropylene tube and closure with glass impinger 1/2- and 1-hour samples use all glass midget impinger.
Critical orifice should be calibrated with rotameter (24-hr sampling) or soap bubble meter or wet test meter (1/2- and 1-hr sampling) or mass flow meter.
All data points within ±2% of the best-fit calibration curve.
Critical orifice should be calibrated before and after each sampling period.
Rotameter cleaned and calibrated every 30 days of operation or every 6 months.
Sample timer should be checked to be accurate within ±15 min/24-hr. every 6 months.</td>
<td>Colorimetric analysis-Blank within ±0.03 absorbance unit of absorbance calibration intercept. Control sample absorbance within ±1.2 μg SO_2 of actual value.
Calibration curve for absorbance vs. μg SO_2/m^3 slope between 0.03 ±0.002 absorbance units/μg SO_2. Intercept below 0.17. All points within 0.8 μg SO_2 of best-fit curve.
Reference samples should be within ±1.2 μg SO_2 of correct value. Any 2 consecutive results outside ±0.8 μg SO_2 in the same direction are unacceptable.</td>
<td>
<table>
<tr><th>Reagent</th><th>Container</th><th>Frequency of preparation</th></tr>
<tr><td>Sulfamic acid, 0.6%</td><td>Glass stoppered</td><td>Prior to analysis</td></tr>
<tr><td>Formaldehyde, 0.2%</td><td>Glass stoppered</td><td>Prior to analysis</td></tr>
<tr><td>Working iodine solution, 0.01 N</td><td>Glass stoppered, dark</td><td>Prior to analysis</td></tr>
<tr><td>Stock iodine solution, 0.1 N</td><td>Glass stoppered, dark</td><td>Yearly</td></tr>
<tr><td>Sodium thiosulfate titrant, 0.01 N</td><td>Glass stoppered</td><td>Yearly</td></tr>
<tr><td>Stock sodium thiosulfate, 0.1 N</td><td>Glass stoppered</td><td>Yearly</td></tr>
</table>
Working sulfite TCM solution shoulo be accurately prepared from standard solution prior to an analysis and is stable for 30 days @ 5°C when stored in a dark glass stoppered bottle.
Standard sulfite TCM solution 320-400 μg/ml SO_2.
Pararosaniline reagent 100% assay is stable when stored in a dark glass stoppered bottle.
Assayed stock pararosaniline solution should be stored in a dark glass stoppered bottle. Prepare new solution when the slope of the calibration curve deviates from 0.03 ± 0.002.
Analytical balance performance should be within ±0.1 mg of manufacturer's specification.
</td>
</tr>
</table>

Figure 5.1B Reverse side laboratory data log

c. Each plotted point must be within 0.8 μg SO_2 units from the best-fit curve. If not:

(1) Repeat the reading for that point.

(2) Average the new and original values for the point.

(3) Plot the average value.

13. File calculations and calibration curve in calibration log book. An example of this calibration curve is presented in Figure 5.2.

14. Determine the calibration factor, B_s (μg SO_2/absorbance unit) which is the reciprocal of the slope.

5.4 Sample Analysis (Twenty-four Hour Sampling)

The following stepwise procedure is presented for analysis of field samples.

1. If no leakage occurred during sample shipment as indicated by the volume as marked on the data sheet after sampling and compared to the sample value as received, dilute the sample with distilled water to 50 ml.

2. If leakage has occurred, invalidate the sample.

3. Pipette 5.0 ml of sample into a 25.0-ml volumetric flask and add 5-ml of absorbing reagent. NOTE: For shorter sampling periods, such as 1 or 3 hours, 10-ml of sample can be used to increase the detection limit, however, the addition of 5-ml of TCM absorbing reagent must be omitted.

4. Delay analyses for 20 minutes to allow for any ozone to decompose.

5. Prepare a reagent blank by pipetting 10.0 ml of absorbing reagent into a 25.0-ml volumetric flask, and carry this blank through the analytical procedure.

6. Prepare an internal control standard by pipetting 2.0 ml of working sulfite-TCM solution and 8.0 ml of absorbing reagent into a 25.0-ml volumetric flask, and carry this control sample through the analytical procedure.

SO_2 Calibration Curve
EPA Reference Method
(Pararosaniline Method)
Analyst Joe Smith Date 3-27-75
Calibration Temperature 25 °C
Calibration Factor (B_s) =

$$(\text{Slope})^{-1} = \frac{1}{0.030} = 33.3\ \mu g\ SO_2/\text{absorbance unit}$$

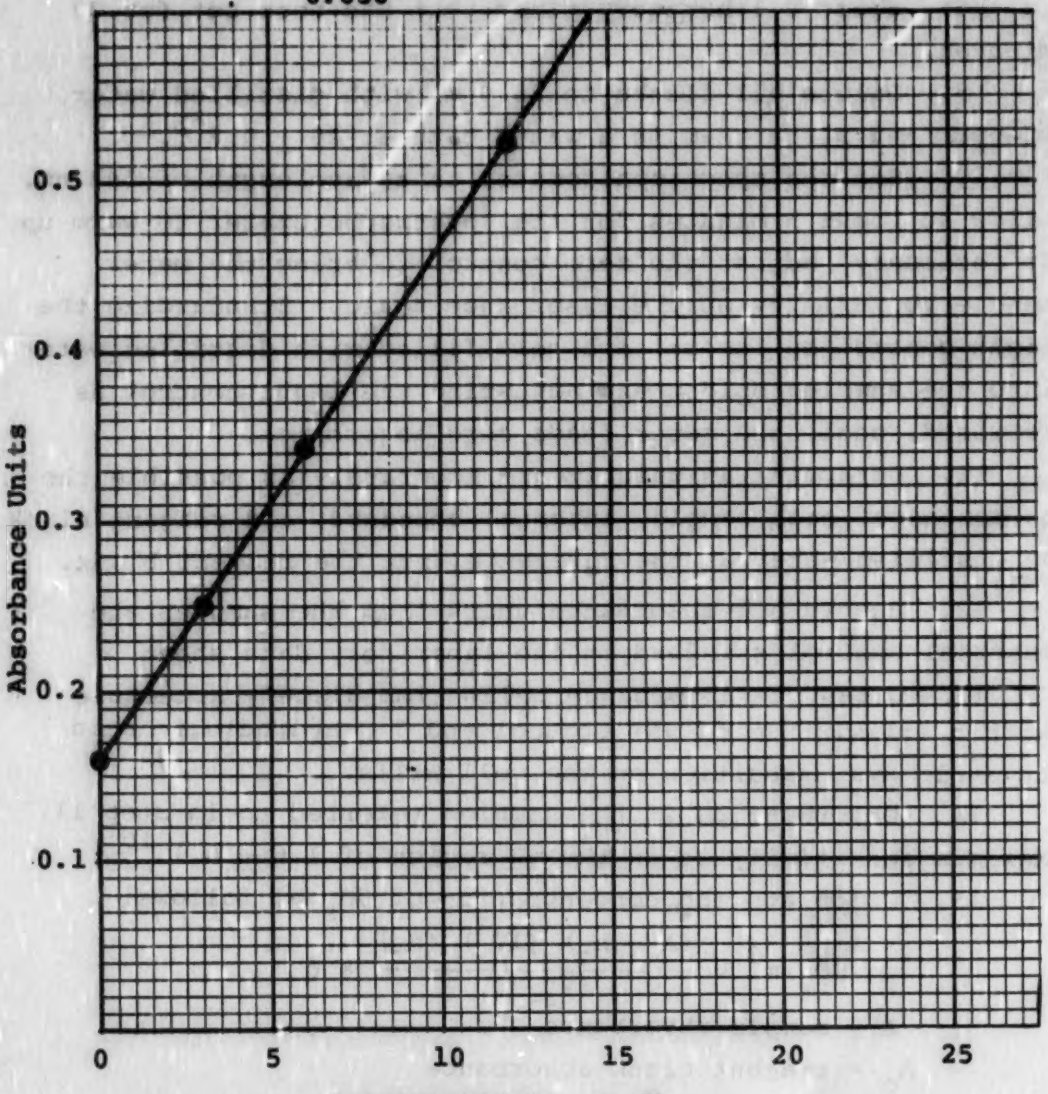


Figure 5.2 Example of SO_2 calibration curve

7. To each 25.0-ml flask add the following:
   a. 1.0 ml of 0.6 percent sulfamic acid and allow to react for 10 minutes to destroy the nitrate from oxides of nitrogen.
   b. 2.0 ml of 0.2 percent formaldehyde solution.
   c. 5.0 ml of pararosaniline solution.

8. Start a laboratory timer that has been set for 30 minutes.

9. Dilute all flasks to 25.0 ml with distilled water, stopper and place them in a water bath at 20 ± 0.2°C.

10. Set the spectrophotometer to a wavelength of 548 nm. Allow at least 5 minutes for the spectrophotometer to warm up. If necessary, adjust the zero control to bring the meter needle to infinity on the absorbance scale. Standardize the light control by inserting a cell filled with distilled water into the sampler holder and adjusting the light control as required, until the meter reads zero absorbance.

11. Determine as quickly and accurately as possible the absorbance of each sample, internal standard, and reagent blank. Use distilled water as the reference, not the reagent blank.

12. Record all absorbance units from the samples and internal control standard on the laboratory data sheet.

13. Clean the absorbance cells immediately after use with a 1:1 mixture of 95% ethanol and 3 N hydrocloric acid to avoid dye deposition on the cell walls.

14. Determine pH of the samples referred to in step 11. Maximum sensitivity is in the pH region of 1.6 ± 0.1.[10]

15. Calculate the concentration of SO_2 as follows:

$$\mu g\ SO_2/m^3 = \frac{(A - A_o)\ (10^3)\ (B_s)}{V_r} \times D$$

A = sample absorbance

A_o = reagent blank absorbance

10^3 = conversion of liters to cubic meters

V_r = the sample air volume corrected to 25°C and 760 mm Hg, liters

B_s = calibration factor, μg/absorbance unit

D = dilution factor (30-min and 1-hr samples, D = 1, and for 24-hr samples, D = 10).

b. Calculation of SO_2 concentration, ppm

$$\text{ppm } SO_2 = \mu g\ SO_2/m^3 \times 3.82 \times 10^{-4}$$

16. If the absorbance of the blank solution is within ±0.03 absorbance units of calibration curve intercept and if the measured value of the internal control sample is within ±1.2 μg SO_2 of the actual value, the analytical values as determined for the field samples can be accepted as valid.

17. If the reagent blank and/or control sample falls outside the above limits the reagent blank, control sample, and calibration curve should be checked by replication to satisfy the above limits before re-analyzing the field samples.

Table 5.1 ACTIVITY MATRIX FOR ANALYSIS OF SAMPLES

Activity	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Verification of field record sheet	All vital information obtained and verified	Visually inspect at end of each sampling period	Obtain missing data or invalidate sample
Flow rate check of systems with critical orifice	Difference in flow rates from initial and final readings after sampling less than 10%	After sampling repeat flow rate measurement	Invalidate the sample
Flow rate check of flow systems with rotameter	Difference in flow rates from initial and final readings from the rotameter calibration curve less than 10%	As above	As above
Calibration of spectrophotometer sample analysis	Each calibration curve: 1) Slope is 0.03 ± 0.002 absorbance unit/μg SO_2 2) Intercept is less than 0.17 absorbance units 3) Each plotted point within 0.8 μg SO_2 units from best-fit curve	After sampling, check the calibration curve	1) Repeat calibration and/or standardize the sulfate solution and check dye 2) Check temperature of water bath and/or check chemicals used in preparing reagents 3) Repeat reading for that point, average new and original values and replot
Sample analysis	Perform steps of analysis in accordance with Section 5.4 1) Absorbance of blank solution is within ±0.03 absorbance units of intercept 2) Measured value of internal control sample is within ±1.2 μg SO_2 of the actual value	Each sample	Check blank, control sample, and calibration curve by replication

6.0 DATA REDUCTION, VALIDATION, AND REPORTING

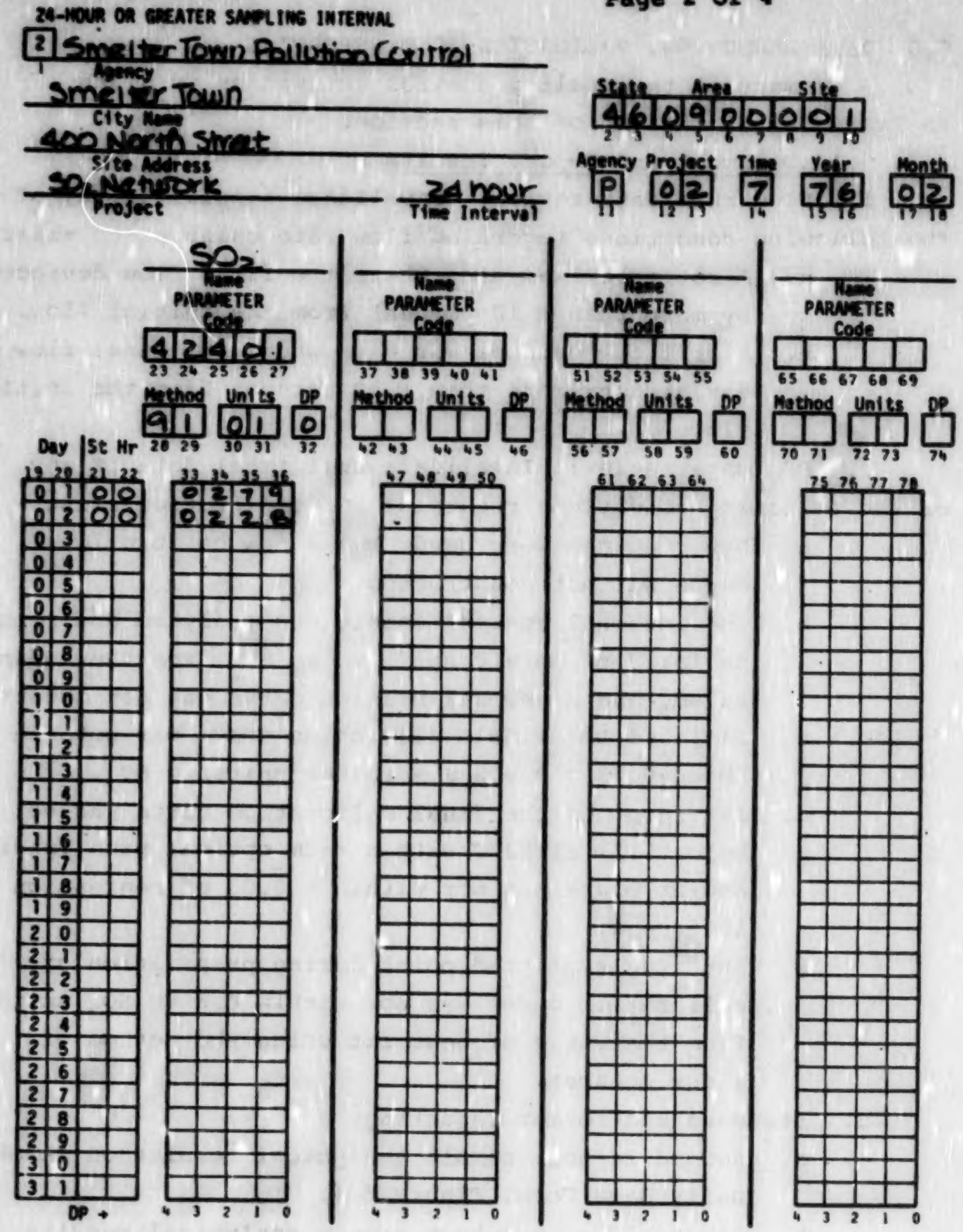
A summary of the quality control activities is given in Table 6.1 at the end of this section.

6.1 Document and Report Results

1. Flow rate data review: Invalidate samples if any of the following conditions regarding flow rate calibration exists.
    a. A rotameter was used where the final flow deviated by more than ± 10 percent from the initial flow.
    b. A critical orifice was used where the final flow deviated by more than ± 10 percent from the initial flow.

2. SO_2 data review: Invalidate analytical data if any of the following conditions regarding SO_2 calibration exist.
    a. New reagents were used, but a new calibration curve was not constructed.
    b. The internal control sample was analyzed and results deviated by more than ± 1.2 μg SO_2 from the known value, and a new calibration curve was not constructed.
    c. Slope of the final calibration curve was not between 0.03 ± 0.002 absorbance units/μg SO_2.
    d. Intercept of the final calibration curve was not below 0.17 at 22°C with a 1-cm optical path length and/or blank was not within ± 0.03 of regression intercept.
    e. Any single plotted point during preparation of calibration curve was not within 0.8 μg SO_2 units from the curve of best fit using the method of least squares.

3. Standardized format reporting.
    a. Record 24-hour sample analytical results on SAROAD Daily Data Form, Figure 6.1.
    b. Record 1/2- or 1-hour sample analytical results on SAROAD Hourly Data Form, Figure 6.2.
    c. Refer to AEROS User's Manual; OAQPS No. 1.2-039, for coding instructions.



24-HOUR OR GREATER SAMPLING INTERVAL

2 (1) Smelter Town Pollution Control
Agency

Smelter Town
City Name

400 North Street
Site Address

SO2 Network
Project

24 hour
Time Interval

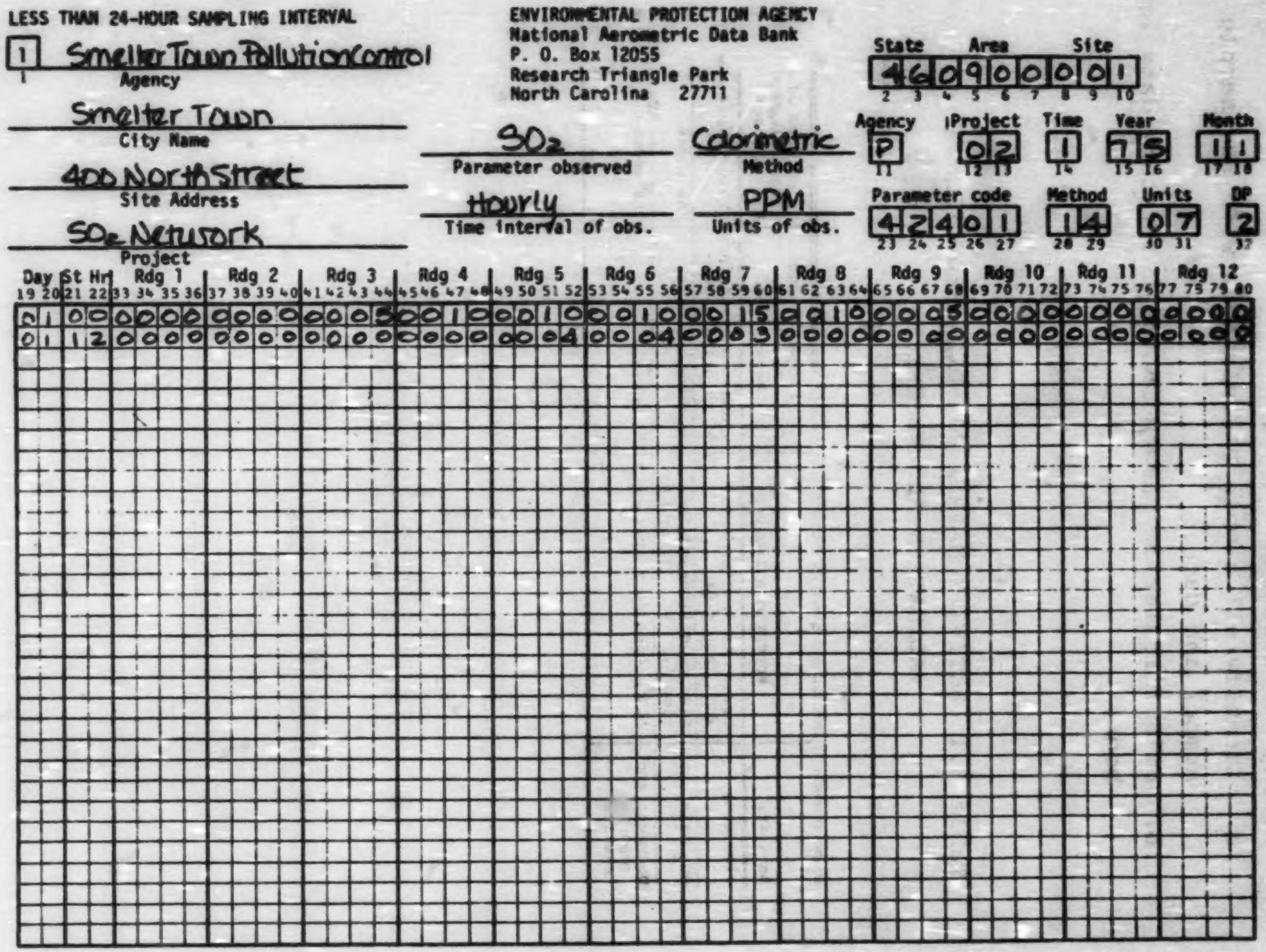
State Area Site: 4 6 0 9 0 0 0 0 1 (2–10)

Agency: P (11)
Project: 0 2 (12 13)
Time: 7 (14)
Year: 7 6 (15 16)
Month: 0 2 (17 18)

Name	SO_2			
PARAMETER Code	4 2 4 0 1 (23 24 25 26 27)	(37 38 39 40 41)	(51 52 53 54 55)	(65 66 67 68 69)
Method	9 1 (28 29)	(42 43)	(56 57)	(70 71)
Units	0 1 (30 31)	(44 45)	(58 59)	(72 73)
DP	0 (32)	(46)	(60)	(74)

Day (19 20)	St Hr (21 22)	33 34 35 36	47 48 49 50	61 62 63 64	75 76 77 78
01	00	0279			
02	00	0228			
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
26					
27					
28					
29					
30					
31					
DP →		4 3 2 1 0	4 3 2 1 0	4 3 2 1 0	4 3 2 1 0

Figure 6.1 SAROAD daily data form.



LESS THAN 24-HOUR SAMPLING INTERVAL

1 Smelter Town Pollution Control
Agency

Smelter Town
City Name

400 North Street
Site Address

SO₂ Network
Project

ENVIRONMENTAL PROTECTION AGENCY
National Aerometric Data Bank
P. O. Box 12055
Research Triangle Park
North Carolina 27711

Parameter observed: SO₂

Method: Colorimetric

Time interval of obs.: Hourly

Units of obs.: PPM

State: 46
Area: 0900
Site: 001
Agency: P
Project: 02
Time: 1
Year: 75
Month: 11
Parameter code: 42401
Method: 14
Units: 07
DP: 2

Day 19 20	St Hr 21 22	Rdg 1 33 34 35 36	Rdg 2 37 38 39 40	Rdg 3 41 42 43 44	Rdg 4 45 46 47 48	Rdg 5 49 50 51 52	Rdg 6 53 54 55 56	Rdg 7 57 58 59 60	Rdg 8 61 62 63 64	Rdg 9 65 66 67 68	Rdg 10 69 70 71 72	Rdg 11 73 74 75 76	Rdg 12 77 78 79 80
01	00	0000	0000	0005	0010	0010	0010	0015	0010	0005	0000	0000	0000
01	12	0000	0000	0000	0000	0004	0004	0003	0000	0000	0000	0000	0000

Figure 6.2 SAROAD hourly data form.

4. Filing calculations:
   a. Identify all sample calculations with the sample number and date.
   b. File calculation sheets in the Laboratory data log book.

Table 6.1 ACTIVITY MATRIX FOR DATA REDUCTION, VALIDATION AND REPORTING

Activity	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Flow data	Deviation in flow rates less than ±10%	Visual observation of reported data	Void sample
SO_2 data	Five conditions of Section 6.1, step 2, are met	As above	As above
Document and report results	All needed data available	As above	As above

7.0 MAINTENANCE

7.1 24-hour Bubbler Train

Every three months or every 15 sampling periods, whichever comes first, replace the following components:

1. Millipore filter - The millipore filter is replaced by removing the Tygon tubing from the inlet and outlet of the plastic filter holder. The filter and filter holder are disposable. The new filter holder is to be sealed to prevent leakage. This is done by pulling the filter holder apart, coating the junction of the two halves with a thin layer of cyclohexanone, and putting the two halves back together and pressing till dry. The new filter is connected back to place.

2. Mist trap - The trap is changed by removing the Tygon tubing and replacing it with a clean, dry trap. The trap that is removed is to be disassembled, washed, and allowed to dry before being reassembled and used again.

3. Rubber septums - Remove old septums from glass adapter and replace with new septums.

4. Vacuum gauge - Check gauge against a calibrated vacuum gauge or mercury manometer. Replace gauge if found defective.

5. Vacuum tubing - Replace black vacuum tubing with new section to eliminate the possibility of leakage.

6. Vacuum pump - If vacuum pump does not have the capability to provide at least 20 in. Hg (508 mm Hg) vacuum at 760 mm atmospheric pressure, replace its carbon vanes or its diaphragm. Twenty inches of Hg (508 mm Hg) vacuum at an atmospheric pressure of 760 mm provides a safety margin of 3.1 in. Hg (79 mm Hg) vacuum. For use under this atmospheric pressure consult Table 2.3.

7.2 Short-term Sampling Train

Every three months or every 30 sampling periods, whichever comes first, maintain the following components:

1. Rotameter - Disassemble and clean rotameters with detergent and rinse with distilled water and alcohol.

2. Mist trap - Clean and replace glass wool.

3. Needle valve - Inspect valve for proper seating of stem and check valve-stem packing nut for proper tension. Clean valve with detergent and rinse with distilled water and alcohol.

4. Vacuum pump - Perform the same maintenance as described in Section 7.1.

5. Vacuum tubing - Perform same maintenance as described in Section 7.1.

7.3 Calibration Equipment and Related Apparatus

Periodic maintenance requirements for such equipment as mass flow meters, soap bubble meters, spectrophotometers, etc. vary with the specific manufacturer and the manufacturer's recommendations should be followed in addition to one's own experience.

Table 7.1 ACTIVITY MATRIX FOR REGULAR AND PREVENTIVE MAINTENANCE

Equipment	Acceptance limits	Frequency and method of measurement	Action if requirements not met
24-hour bubbler train	Components replaced: millipore filter, mist trap, rubber septums, and vacuum tubing	Replace every 3 months or after 15 sampling periods	Implement maintenance actions
Vacuum gauge	Correct readings within ± 1 inch (25.4 mm) of Hg	Check every 3 months against calibrated vacuum gauge or Hg manometer	Replace if defective
Vacuum pump	Capability to provide at least 30 inch (508 mm) Hg vacuum at STP	Vacuum test	Replace carbon vanes or diaphragm and retest
Short-term sampling train	Maintain components: rotameter, mist trap, needle valve, vacuum pump, and vacuum tubing	Clean every 3 months or after 30 sampling periods	Implement maintenance actions
Calibration equipment and related apparatus	Periodic maintenance performed according to recommendations	Follow manufacturer's recommendations for maintenance	Implement maintenance actions

8.0 AUDITING PROCEDURE

Audit as used here implies an independent assessment of the quality of data obtained by the ambient air monitoring methods. Independence can be achieved by having the audit made by a different operator/analyst than the one conducting the routine field measurements. The audit should be a true assessment of the measurement process under normal operation, i.e., without any special preparation or adjustment of the system. Routine quality assurance checks conducted by the operator/analyst are necessary for obtaining and reporting good quality data, but they are not to be considered as part of the auditing procedure.

Two types of audits are recommended herein, performance and systems audits. Three performance audits and a systems audit are described in detail in the following sections. An alternative to performing three individual audits is to audit the entire measurement process by comparing the final results from the field sampler to those obtained with a collocated sampler. This alternative is described in Section 8.2. A summary of these audits is given in Table 8.3 at the end of this section. A systems audit is described in Section 8.3.

Proper implementation and conduct of an auditing program will serve a two-fold purpose: (1) to screen the data, by lots of say N = 50 to 100 measurements of concentration of SO_2 to detect whether the data quality are adequate, and (2) to estimate data quality in terms of the precision and accuracy. Appendices G and I of Volume I of this Handbook[8] contain information for the auditing and sampling procedures presented in this section and techniques for estimating precision and accuracy of the data.

8.1 Performance Audits

Performance audits are independent assessments made by another operator/analyst to evaluate the quality of data produced by the total measurement system (sample collection, sample analysis, and data processing). Performance audits are normally a quantitative appraisal of quality.

Three performance audits of individual variables are recommended:

1. Audit of flow rate for sample collection.
2. Audit of analysis process using reference samples.
3. Audit of data processing.

An auditing level of 7 out of 100 sampling periods for each site is suggested here as a starting frequency. For the case where one sample is collected every sixth day, an auditing level of one per month per site is recommended. This would result in an auditing level of approximately 3 for each 15 sampling periods when the data are reported quarterly. If the number of sampling periods is greater than 15 but less than 50, four (4) audits are recommended. These frequencies are suggested starting frequencies and they are to be altered based on experience and data quality. The audit frequency should be reduced if past experience indicates the data are of good quality, the converse holds if the data are of poor quality. In determining the number of audits to be made for a large lot size, it is more important to make sure that the audit sample is representative of the various conditions that may influence data quality than to adhere to a fixed frequency. The supervisor will specify the audits and auditing levels to be used according to monitoring requirements.

8.1.1 Flow Rate Audit - For 30-minute, 1-hour and 24-hour samples, a flow rate audit is recommended as a means of assessing the sample collection phase of the measurement process. The audit is performed as follows:

1. The regular operator prepares the sampler for sample collection as usual. This includes filling in the Sample Record Sheet, Figure 4.9.

2. Compute the average sampled volume, $\overline{V}_m$, measured by the regular rotameter and/or critical orifice and corrected to reference conditions according to equation 2-4, Section 2.1.2.

3. At the beginning and at the end of the sampling period, the auditor inserts the audit rotameter in the sample inlet line and a flow measurement is made in the usual manner (see Section 2.1.5).

4. Calculate the average audited volume, $\overline{V}_a$, at reference conditions for the audit rotameter, $\overline{V}_a = \overline{Q}_a t$, where $\overline{Q}_a$ is the average audited flow rate in liters/minute and t is the sampling period time in minutes. See equation 2-4, Section 2.1.4, for correction to reference conditions.

5. Compute the percent accuracy, %A, by

$$\%A = \frac{\overline{V}_m - \overline{V}_a}{\overline{V}_a} \times 100 \qquad \text{Equation 8-1}$$

6. Record $\overline{V}_m$, $\overline{V}_a$ and %A and plot the %A's on the $\overline{X}$ and R chart as illustrated in Figure 8.1.[a]

7. If %A is greater than ± 7 percent or $\overline{V}_m/\overline{V}_a$ does not fall between 0.93 and 1.07, trouble shooting should be performed and corrective action taken before sampling is resumed. All flow rate data for the audit period is invalidated.

8.1.2 Audit of Analysis Process Using Reference Samples - Reference samples should be prepared using (1) a working Sulfite-TCM solution (see Section 6 of the Reference Method) that was prepared from the same reagent but independently from the regularly used solution. Reference samples should span a range of 0.20 to 1.0 absorbance units. The specific values should vary from audit to audit. The measurement of the independent reference samples evaluates the overall analysis procedure including operator, chemicals, and technique.

Reference (audit) samples should be dispersed randomly throughout the samples awaiting analysis at the recommended frequency. If possible, they should not be recognizable to the analyst as a reference sample.

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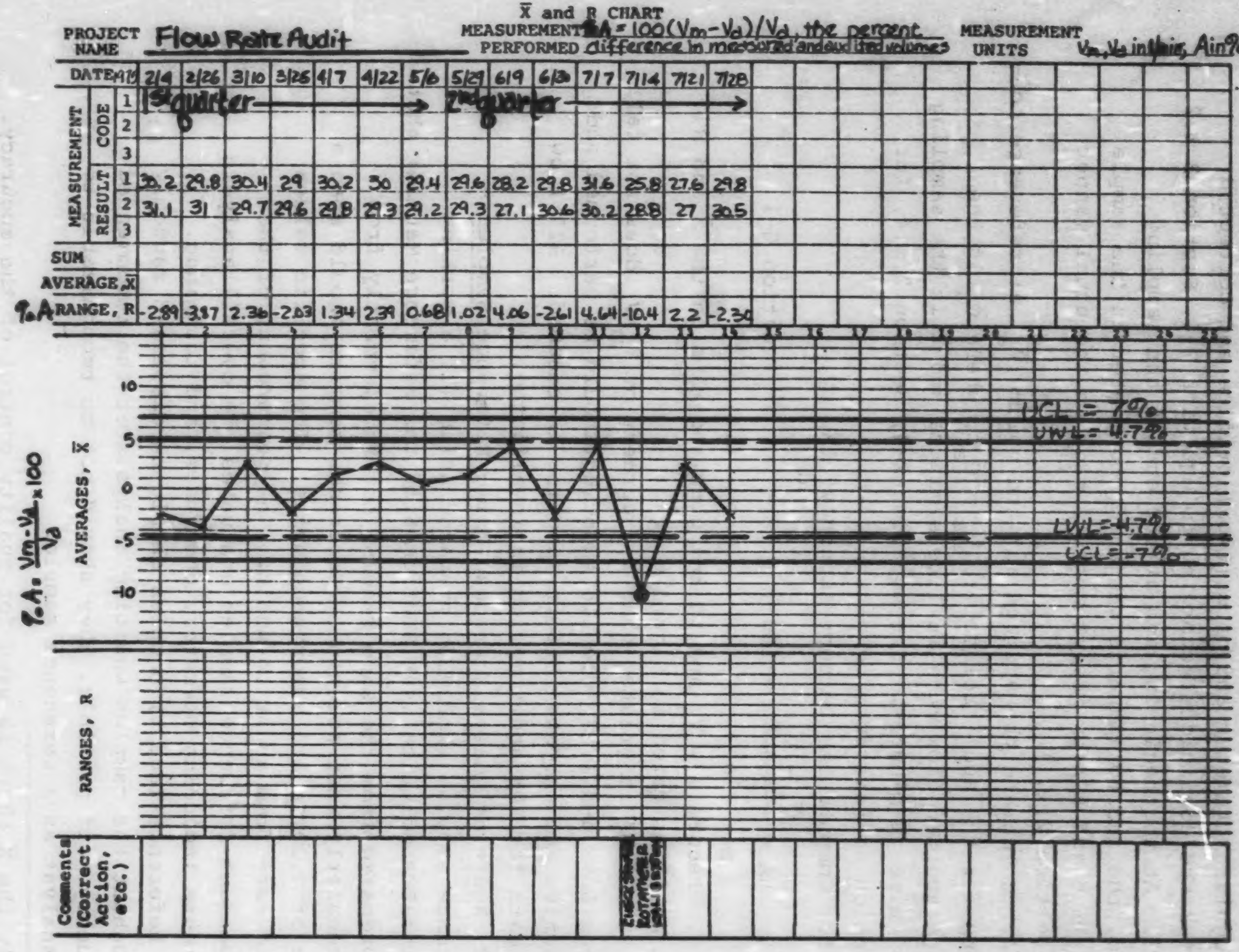
[a] Only the $\overline{X}$ chart is used for quality control of the accuracy. The Standard $\overline{X}$ and R chart is used for convenience.



PROJECT NAME Flow Rate Audit

MEASUREMENT PERFORMED %A = 100(Vm−Va)/Va, the percent difference in measured and audited volumes

MEASUREMENT UNITS Vm, Va in l/min, A in %

DATE 1979		2/4	2/26	3/10	3/26	4/7	4/22	5/6	5/29	6/19	6/30	7/7	7/14	7/21	7/28
MEASUREMENT CODE	1	1st quarter →							2nd quarter →						
	2														
	3														
MEASUREMENT RESULT	1	30.2	29.8	30.4	29	30.2	30	29.4	29.6	28.2	29.8	31.6	25.8	27.6	29.8
	2	31.1	31	29.7	29.6	29.8	29.3	29.2	29.3	27.1	30.6	30.2	28.8	27	30.5
	3														
SUM															
AVERAGE, X̄															
%A RANGE, R		-2.89	-3.87	2.36	-2.03	1.34	2.39	0.68	1.02	4.06	-2.61	4.64	-10.4	2.2	-2.30

Figure 8.1 Example of an $\overline{X}$ and R chart
Audit of flow rate using % A

1. Prepare a reference sample with a known concentration in the same manner as was used when developing a calibration curve. The concentration should be varied over the range of normally measured values from audit to audit.

2. Have the regular operator determine the absorbance of the reference sample. The operator must not know the true concentration of the reference sample and preferably not know that it is a reference sample.

3. Obtain the operator's finding ($\mu g\ SO_2)_m$ and take the audit value ($\mu g\ SO_2)_a$ for the sample and compute the accuracy by

$$A = (\mu g\ SO_2)_m - (\mu g\ SO_2)_a. \qquad \text{Equation 8-2}$$

4. Record ($\mu g\ SO_2)_m$, ($\mu g\ SO_2)_a$ and A on the $\overline{X}$ and R chart for each audit as illustrated in Figure 8.2.

5. It is recommended that the analysis be stopped, checks made to determine the assignable cause(s), and corrective action taken before the analysis is resumed anytime:

   a. A value of A numerically equal to or greater than 1.2 $\mu g\ SO_2$ is observed, or
   b. Two A values out of three consecutive audits exceed ± 0.8 $\mu g\ SO_2$ in the same direction, i.e., have the same sign and exceed 0.8 in numerical value.

Results from a collaborative test of the pararosaniline method show that precision is a function of the SO_2 concentration.[2,3,4] The performance standard given herein for measurement of reference samples represents the 3σ value from that test. This standard should be reevaluated and adjusted, if necessary, as field data become available.

6. If the reference sample measurement is outside the control limits, repeat the check employing another analyst. If the results of the second check are outside the control limits, the calibration curve should be checked by measuring a reagent blank and control sample. If necessary, a new calibration

$\overline{X}$ and R CHART

PROJECT NAME Measurement of Audit (Reference) Samples

MEASUREMENT PERFORMED A = (μg SO₂)ₘ − (μg SO₂)ₐ = difference between operator's finding and known or audited value

MEASUREMENT UNITS μg SO₂

DATE	1975	2/4	2/26	3/10	3/26	4/7	4/22	5/6	5/29	6/19	6/30	7/7	
MEASUREMENT CODE	1	1st Quarter →							2nd Quarter →				
	2												
	3												
MEASUREMENT RESULT	1	5	2	7	5	6	9	4	7	8	3	10	= (μg SO₂)ₘ
	2	4.7	1.9	7	4.9	6.1	9.2	3.4	7.5	7.8	2.3	10.2	= (μg SO₂)ₐ
	3												
SUM													
AVERAGE, $\overline{X}$													
A =		.3	.1	0	.10	−.1	−.2	.6	−.5	.2	.7	−.2	

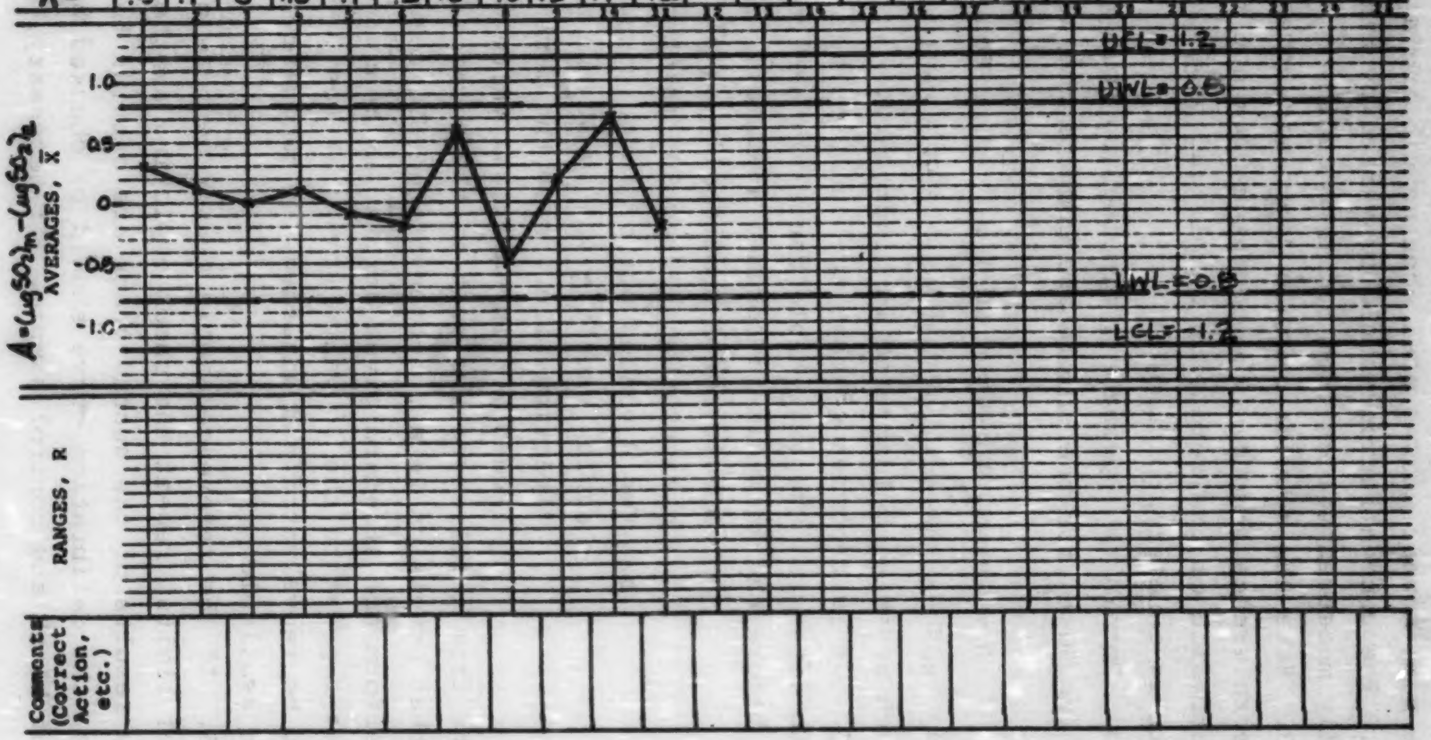


Figure 8.2 Example of an $\overline{X}$ and R chart for SO_2 reference samples

curve should be developed and other corrective actions taken as deemed necessary.

7. After corrections are made, a new reference sample should be measured and, if acceptable, analysis resumed.

8. If the reference sample measurement is within limits outlined above, continue normal operations.

8.1.3 Data Processing Audit - In auditing data processing procedures, it is convenient if audits are made soon after the original calculations have been performed. This allows corrections to be made immediately and for possible retrieval of additional explanatory data from field personnel when necessary. The procedural steps follow:

1. The audit must be independent; that is, performed by an individual other than the one who originally reduced the data. The check is made starting with the raw data and continuing through the recording of the concentration in $\mu g/SO_2/m^3$ on the SAROAD form.

2. If the mass concentration of SO_2 computed by an audit differs from the original value by more than ± 3 percent, i.e., if

$$\%A = \frac{(\mu g\ SO_2/m^3)_m - (\mu g\ SO_2/m^3)_a}{(\mu g\ SO_2/m^3)_a} \times 100 \qquad \text{Equation 8-3}$$

is larger than 3 in absolute value, then all samples collected since the previous audit are checked and corrected. The audit value is always reported as the correct value under the assumption that if a discrepancy exists, the audit should be checked and, if necessary, corrected.

3. Record the audit and original values and the percent difference in the laboratory log book and report the values to the supervisor.

8.2 Auditing with a Collocated Sampler

An alternative method of auditing the SO_2 Reference Method is to use a collocated sampler as a reference. The use of an independent sampling train provides not only a means of auditing the data but also an assessment of the data quality. The reliability of this procedure is directly dependent on how well the collocated sampler is maintained, the accuracy/precision of the sample analysis procedures, the environmental concentration of SO_2, and on the auditor. The sampler should be operated in strict accordance with the procedures given in this section of the Handbook. A record should be maintained of the checks and audits performed on the sampler and reported with the data as requested by the manager.

Based on the results of a series of tests at several concentrations of SO_2 under well-controlled conditions in an EPA laboratory, the mean and standard deviation of SO_2 concentrations for samples collected simultaneously by five sampling trains using a common manifold are given in the first two columns of Table 8.1. These results indicate that the standard deviation, s, is dependent upon the average SO_2 concentration, $\bar{X}$, for the five trains. The relative standard deviation (RSD) (or coefficient of variation) is computed and given in the third column of Table 8.1; $RSD = 100s/\bar{X}$. Columns 4 and 5 in Table 8.1 are obtained by multiplying the values in columns 2 and 3 by $3\sqrt{2}$. These values are the expected agreement between two collocated samplers on the same manifold under well-controlled conditions. They estimate 3σ control limits corresponding to values that should include about 99.7 percent of the differences between the two samples under "ideal" laboratory conditions. The 3σ limits expressed as a percentage of the average concentration, $\bar{X}$, values in the last column of Table 8.1, are plotted in Figure 8.3. A smooth curve is drawn through the data points with a minimum near 365 µg SO_2/m^3. The values read from the smooth curve would be the recommended 3σ limits under best conditions of sampling and analysis.

Table 8.1 EXPERIMENTAL DATA AND EXPECTED AGREEMENT BETWEEN COLLOCATED (TWO) SO_2 SAMPLING TRAINS ON A COMMON MANIFOLD UNDER WELL-CONTROLLED CONDITIONS

Experimental data[a]			Expected agreement between collocated (two) sampling trains[b]	
			3σ control limits expressed as:	
Measured average concentration ($\overline{X}$) µg SO_2/m^3	Standard deviation of measured concentration (s) µg SO_2/m^3	Relative standard deviation $\left[\frac{100s}{\overline{X}}\right]$	µg SO_2/m^3	Percent of the average concentration %
16.7	2.53	15.1	10.7	64
35.6	4.52	12.7	19.2	54
37.8	3.13	8.3	13.3	35
71.2	4.54	6.4	19.3	27
72.4	1.17	1.6	5.0	7
73.9	3.43	4.6	14.6	20
177	5.81	3.3	24.6	14
201	3.37	1.7	14.3	7
344	2.61	0.8	11.1	3
684	17.8	2.6	75.5	11
694	27.1	3.9	115	17
927	44.9	4.8	190	20

[a] Based on experimental data for 24-hour samples obtained by a single analyst using five trains on a common manifold (Fuerst, R. G. Effect of Temperature on Stability of Sulfur Dioxide Samples. Unpublished research by the Environmental Monitoring and Support Laboratory, Research Triangle Park, North Carolina, March 1976).

[b] The results in columns 4 and 5 are calculated by multiplying the results in column 2 and 3, respectively, by $3\sqrt{2}$. These data are plotted in Figure 8.3 and an eye-fitted smooth curve is drawn to provide 3σ control limits which represent the best that might be expected for agreement for collocated samplers using the SO_2 reference method.

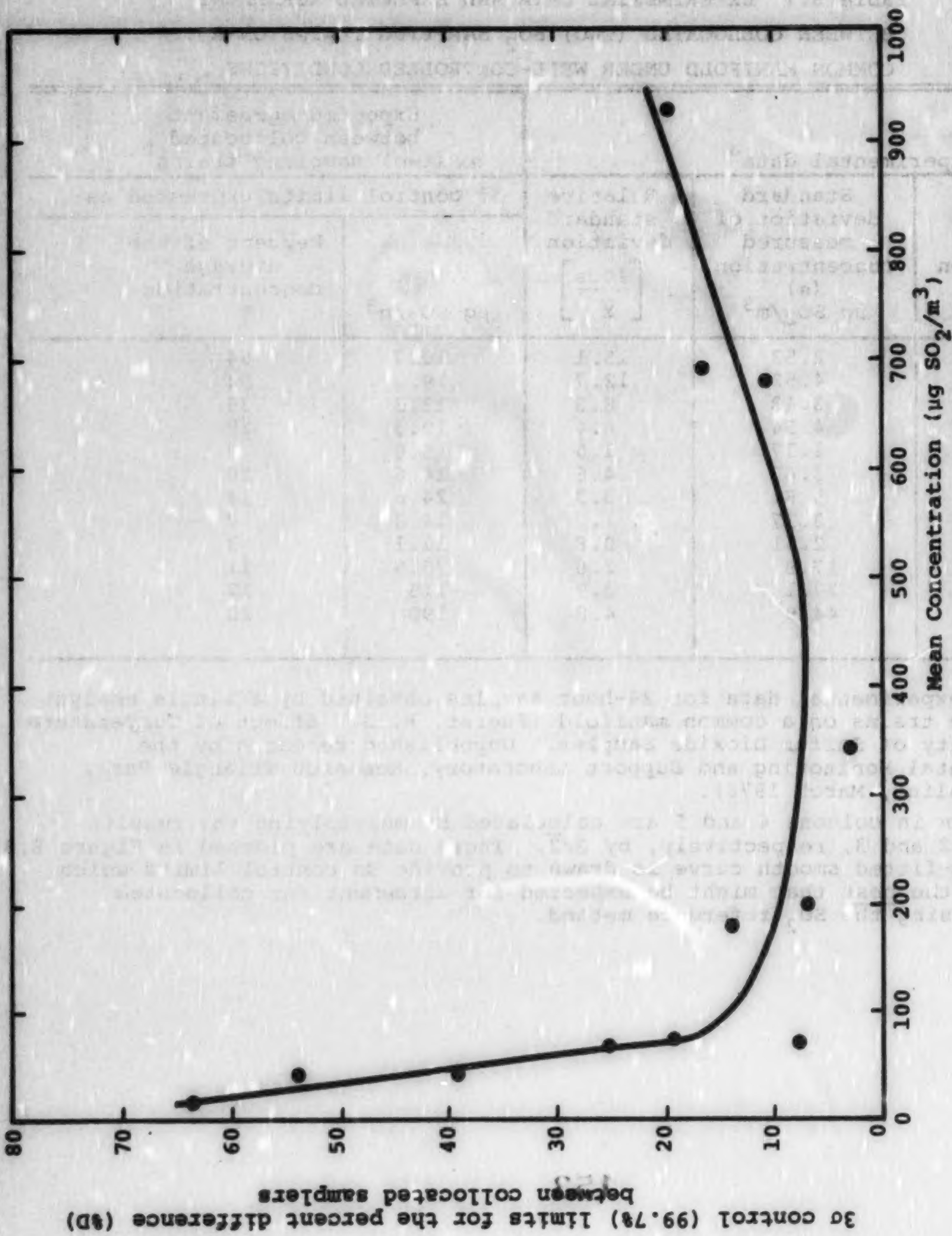


Figure 8.3 Agreement between collocated (two) SO$_2$ sampling trains under well-controlled conditions (Based on laboratory experiment data in Table 8.1)

Actual field data for collocated samplers show a similar trend to that in Figure 8.3. However the 3σ limits are much larger. For concentrations exceeding 75 μg SO_2/m^3, the 3σ limits of agreement are about 45 percent of the average concentration. For low concentrations these limits often exceed 100 percent.

Based on the experimental data in Table 8.1 and the field test data, collocated sampling is recommended only when the environment to be sampled is expected to have an SO_2 concentration exceeding 75 μg SO_2/m^3. At these concentrations the 3σ limits of agreement between collocated SO_2 sampling trains should be between about 15 percent and 45 percent of the average concentration. These limits would be expected to include at least 99.7 percent of the percent differences (%D) as computed by

$$\%D = \frac{(\mu g\ SO_2/m^3)_f - (\mu g\ SO_2/m^3)_a}{0.5\left[(\mu g\ SO_2/m^3)_f + (\mu g\ SO_2/m^3)_a\right]} \times 100, \quad \text{Equation 8-4}$$

where the subscripts f and a refer to the concentrations of the field and audit samples, respectively. In order to detect any trends or unusually large differences that may occur in the results of collocated samplers, the values of %D should be recorded on an $\overline{X}$ and R chart. The 3σ limits of agreement should be determined by the user and they are expected to be between 15 and 45 percent of the average concentration.

8.3 Systems Audit

A systems audit is an on-site inspection and review of the quality assurance system used for the total measurement system (sample collection, sample analysis, data processing, etc.). System audits are normally a qualitative appraisal of system quality.

A systems audit should be conducted at the start up of new monitoring system and as appropriate thereafter to audit significant changes in system operation.

A preliminary form for use in a systems audit is given in Table 8.2. These questions should be checked for the applicability to the particular local, state, or Federal agency.

Table 8.2 CHECK LIST FOR USE BY AUDITOR FOR SO_2 METHOD

1. What type of manual sampler is used for the collection of SO_2? (a) RAC, (b) 5-port, (c) 2-port sampler, (d) other.

2. Is a straight tube impinger used in the polypropylene sampling tube? ____

3. What is the method of analysis?______________________________
Is a copy available? ____

*This method should be taken from the CFR 40, Part 50, July 1975.

4. What quality of reagents is being used to make up the chemicals for the analysis of SO_2? ______________________________

*They should be at least reagent grade, or better.

5. Is the sampling train routinely checked for leaks? ____
How often? (a) once a week, (b) once a month, (c) once a quarter.

6. What calibration procedure is utilized for the field samplers? ______________. Is there a written copy available? ______

7. Are the hypodermic needles calibrated before and after the sampling period? ____. Are they shipped back to the laboratory for calibration in the sample block? ____. Is there a procedure established to discard needles that are not within certain flow rate limits initially? ____. If so, what are the flow rate limits? ________.

*Any needle that is found outside of 180 to 220 cc per minute should be discarded. If it is found to be outside of this flow rate after the sampling period, this analysis should be voided.

8. Are calibration curves made, and are they available? _______
Is the calibration history of the analyses to be found in a bound notebook, or other type of notebook, and is it readily available? ______________________________

*It is important that all laboratories keep a bound notebook indicating when the curves were made, the points used to make the curves, and all in all keep a permanent record of the calibration history.

9. Are collected samples delivered from the field to the laboratory in a container to prevent crushing, spillage, etc.? ____

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* See bottom of Table for comment.

Table 8.2 (continued) CHECK LIST FOR USE BY AUDITOR

10. How much time (in days) is there between sampling and analysis? ____days.

*A common time between sampling and analysis is approximately 12 days. Any time less than this is very good; any time greater than this may cause problems in analysis.

11. Are samples stored in the dark, once they have been collected, until the time of analysis? ____. At what temperature are the samples stored? ____°C.

12. What calculations are done to obtain the final concentration of SO_2? ____________________________

*If possible observe sample calculations that have been completed.

13. How are discrepancies in data treated?

*Is there some format for taking care of discrepancies found in the data?

14. Are the data reported on a quarterly basis? ____. If not, how often? ____________________________

____

*Comments concerning answers to questions and/or remarks to the auditor.

Table 8.3 ACTIVITY MATRIX FOR AUDITING PROCEDURE

Audit	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Flow rate	$0.93 \le \frac{V_m}{V_a} \le 1.07$, where V_m = routinely measured sample volume V_a = audited volume	Frequency: 1 in 14 days of sampling or 1/mo., whichever is greater Method: Calibrated rotameter	Take corrective action before sampling is resumed. Note action on $\bar{X}$ and R chart
Reference samples	$(\mu g\ SO_2)_a = (\mu g\ SO_2)_m \pm 1.2\ \mu g\ SO_2$ where $(\mu g\ SO_2)_a$ = known value for reference sample $(\mu g\ SO_2)_m$ = routinely measured value for reference sample	Frequency: Randomly disperse reference samples among field samples at a rate of 7 per 100. Method: Analyze reference samples using same method as for routine field samples.	Calibration curve should be checked, if necessary. A new reference sample checked and if acceptable, analysis resumed. Note action on $\bar{X}$ and R chart.
Data processing	$0.97 \le \frac{(\mu g\ SO_2/m^3)_m}{(\mu g\ SO_2/m^3)_a} \le 1.03$ where $(\mu g\ SO_2/m^3)_m$ = reported concentration $(\mu g\ SO_2/m^3)_a$ = audited concentration	Frequency: 1 in 14 samples or 1/mo., whichever is greater Method: Independent analysis starting with raw data through the final recorded data	All samples collected since the previous audit are checked and corrected
Collocated sampler audit[a]	Under well-controlled lab conditions, %D should be less than values given in Figure 8.3, where $\%D = \frac{[(\mu g SO_2/m^3)_f - (\mu g SO_2/m^3)_a]100}{0.5[(\mu g SO_2/m^3)_f + (\mu g SO_2/m^3)_a]}$ The subscripts f and a refer to the 2 samples. Under field conditons %D should be less than 45% of the average concentration of the 2 samples.	Frequency: 1 in 14 samples or 1/mo. at sites where the expected environmental SO_2 concentration exceeds 75 $\mu g/m^3$ Method: Use collocated samplers and independent analysis	Take corrective action before further sampling. Note action on $\bar{X}$ and R chart
Systems audit	Method as described in this section of the Handbook	Frequency: At the beginning of a new monitoring system and periodically as appropriate Method: Observation of procedures and by check list	Initiate improved methods and/or training programs

9.0 RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY

To achieve data of desired quality, two essential considerations are necessary, (1) the measurement process must be in a state of statistical control at the time of the measurement and (2) the systematic errors, when combined with the random variation (errors of measurement), must result in a suitably small uncertainty.[11] As evidence in support of good quality data it is necessary to perform quality control checks and independent audits of the measurement process, to document these data by means of a quality control chart, for example, as described in Section 8 for audits, and to use materials, instruments, and measurement procedures that can be traced to an appropriate standard of reference.

Data must be routinely obtained by repeat measurements of standard reference samples, primary, secondary and/or working standards and the establishment of a condition of process control. The working calibration standards should be traceable to standards of higher accuracy, such as those listed in Table 9.1.

Table 9.1 LIST OF RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY

1. Class "S" weights (made to NBS specifications) are recommended for the analytical balance calibration. See Section 2.3 for details on balance calibration checks.
2. SO_2 permeation tubes should be traceable to NBS - Standard Reference Materials - Numbers 1625-1627.[12] See Section 10, subsection 8.2.2 for details of calibration using SO_2 permeation tubes.
3. Reagents should be at least ACS reagent grade - chemicals that have been certified to contain impurities in concentrations below the specifications of the committee of analytical reagents of the American Chemical

Society. Each bottle is identified by batch number.

4. The complete absorbance scale should be checked with a calibrated set of filters, SRM's 2101-2105[12] from the NBS, periodically and anytime a control sample cannot be measured with ± 1.2 μg SO_2 of its known value. See sections 5.3 and 5.4 for details of calibration of spectrophotometer.

5. The spectrophotometer should be checked for proper wavelength calibration throughout the range of the instrument. Chemical solutions with known wavelengths are commercially available from laboratory suppliers. The solutions are to be scanned on the spectrophotometer to determine the maximum absorbance for each specific chemical solution. The absorption is plotted versus wavelength to determine the maximum absorbance for the specific solution. Comparison of the maximum absorbance to the spectrophotometer wavelength dial should agree within ± 0.02 Optical Density. Should deviations exceed ± 0.05 Optical Density, remedial action should be taken.

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[a] Harleco, Philadelphia, Pennsylvania and Oxford Laboratories, Foster City, California are two suppliers.

10.0 REFERENCE METHOD *

APPENDIX A—REFERENCE METHOD FOR THE DETERMINATION OF SULFUR DIOXIDE IN THE ATMOSPHERE (PARAROSANILINE METHOD)

1. *Principle and Applicability.* 1.1 Sulfur dioxide is absorbed from air in a solution of potassium tetrachloromercurate (TCM). A dichlorosulfitomercurate complex, which resists oxidation by the oxygen in the air, is formed (*1, 2*). Once formed, this complex is stable to strong oxidants (e.g., ozone, oxides of nitrogen). The complex is reacted with pararosaniline and formaldehyde to form intensely colored pararosaniline methyl sulfonic acid (*3*). The absorbance of the solution is measured spectrophotometrically.

1.2 The method is applicable to the measurement of sulfur dioxide in ambient air using sampling periods up to 24 hours.

2. *Range and Sensitivity.* 2.1 Concentrations of sulfur dioxide in the range of 25 to 1,030 μg/m.^{3} (0.01 to 0.40 p.p.m.) can be measured under the conditions given. One can measure concentrations below 25 μg./m.^{3} by sampling larger volumes of air, but only if the absorption efficiency of the particular system is first determined. Higher concentrations can be analyzed by using smaller gas samples, a larger collection volume, or a suitable aliquot of the collected sample. Beer's Law is followed through the working range from 0.03 to 1.0 absorbance units (0.8 to 27 μg. of sulfite ion in 25 ml. final solution computed as SO_2).

2.2 The lower limit of detection of sulfur dioxide in 10 ml. TCM is 0.75 μg. (based on twice the standard deviation) representing a concentration of 25 μg./m^3 SO_2 (0.01 p.p.m.) in an air sample of 30 liters.

3. *Interferences.* 3.1 The effects of the principal known interferences have been minimized or eliminated. Interferences by oxides of nitrogen are eliminated by sulfamic acid (*4, 5*), ozone by time-delay (*6*), and heavy metals by EDTA (ethylenediaminetetraacetic acid, disodium salt) and phosphoric acid (*4, 6*,). At least 60 μg. Fe (III), 10 μg. Mn(II), and 10 μg. Cr(III) in 10 ml. absorbing reagent can be tolerated in the procedure. No significant interference was found with 10 μg. Cu (II) and 22 μg. V(V).

4. *Precision, Accuracy, and Stability.* 4.1 Relative standard deviation at the 95 percent confidence level is 4.6 percent for the analytical procedure using standard samples. (*5*)

4.2 After sample collection the solutions are relatively stable. At 22° C. losses of sulfur dioxide occur at the rate of 1 percent per day. When samples are stored at 5° C. for 30 days, no detectable losses of sulfur dioxide occur. The presence of EDTA enhances the stability of SO_2 in solution, and the rate of decay is independent of the concentration of SO_2. (*7*)

5. *Apparatus.*

5.1 *Sampling.*

5.1.1 *Absorber.* Absorbers normally used in air pollution sampling are acceptable for concentrations above 25 μg./m.^{3} (0.01 p.p.m.). An all-glass midget impinger, as shown in Figure A1, is recommended for 30-minute and 1-hour samples.

For 24-hour sampling, assemble an absorber from the following parts:

Polypropylene 2-port tube closures, special manufacture (available from Bel-Art Products, Pequannock, N.J.).

Glass impingers, 6 mm. tubing, 6 inches long, one end drawn to small diameter such that No. 79 jewelers drill will pass through, but No. 78 jewelers drill will not. (Other end fire polished.)

Polypropylene tubes, 164 by 32 mm. (Nalgene or equal).

5.1.2 *Pump.* Capable of maintaining an air pressure differential greater than 0.7 atmosphere at the desired flow rate.

5.1.3 *Air Flowmeter or Critical Orifice.* A calibrated rotameter or critical orifice capable of measuring air flow within ±2 percent. For 30-minute sampling, a 22-gauge hypodermic needle 1 inch long may be used as a critical orifice to give a flow of about 1 liter/minute. For 1-hour sampling, a 23-gauge hypodermic needle five-eighths of an inch long may be used as a critical orifice to give a flow of about 0.5 liter/minute. For 24-hour sampling, a 27-gauge hypodermic needle three-eighths of an inch long may be used to give a flow of about 0.2 liter/minute. Use a membrane filter to protect the needle (Figure A1a).

5.2 *Analysis.*

5.2.1. *Spectrophotometer.* Suitable for measurement of absorbance at 548 nm. with an effective spectral band width of less than 15 nm. Reagent blank problems may occur with spectrophotometers having greater spectral band width. The wavelength calibration of the instrument should be verified. If transmittance is measured, this can be converted to absorbance:

$$A = \log_{10}(1/T)$$

6. *Reagents.*

6.1 *Sampling.*

6.1.1 *Distilled water.* Must be free from oxidants.

6.1.2 *Absorbing Reagent* [*0.04 M Potassium Tetrachloromercurate* (*TCM*)]. Dissolve 10.86 g. mercuric chloride, 0.066 g. EDTA (ethylenediaminetetraacetic acid, disodium salt), and 6.0 g. potassium chloride in water and bring to mark in a 1,000-ml. volumetric flask. (Caution: highly poisonous. If spilled on skin, flush off with water immediately). The pH of this reagent should be approximately 4.0, but it has been shown that there is no appreciable difference in collection efficiency over the range of pH 5 to pH 3.(*7*) The absorbing reagent is normally stable for 6 months. If a precipitate forms, discard the reagent.

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*Taken from CFR 40, Protection of the Environment, Part 50.11, July 1, 1975.

$$\frac{10^3 \text{(conversion of g. to mg.)} \times 0.1 \text{ (fraction iodate used)}}{35.67 \text{ (equivalent weight of potassium iodate)}}$$

6.2 *Analysis.*

6.2.1 *Sulfamic Acid (0.6 percent).* Dissolve 0.6 g. sulfamic acid in 100 ml. distilled water. Prepare fresh daily.

6.2.2 *Formaldehyde (0.2 percent).* Dilute 5 ml. formaldehyde solution (36-38 percent) to 1,000 ml. with distilled water. Prepare daily.

6.2.3 *Stock Iodine Solution (0.1 N).* Place 12.7 g. iodine in a 250-ml. beaker; add 40 g. potassium iodide and 25 ml. water. Stir until all is dissolved, then dilute to 1,000 ml. with distilled water.

6.2.4 *Iodine Solution (0.01 N).* Prepare approximately 0.01 N iodine solution by diluting 50 ml. of stock solution to 500 ml. with distilled water.

6.2.5 *Starch Indicator Solution.* Triturate 0.4 g. soluble starch and 0.002 g. mercuric iodide (preservative) with a little water, and add the paste slowly to 200 ml. boiling water. Continue boiling until the solution is clear; cool, and transfer to a glass-stoppered bottle.

6.2.6 *Stock Sodium Thiosulfate Solution (0.1 N).* Prepare a stock solution by dissolving 25 g. sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in 1,000 ml. freshly boiled, cooled, distilled water and add 0.1 g. sodium carbonate to the solution. Allow the solution to stand 1 day before standardizing. To standardize, accurately weigh, to the nearest 0.1 mg., 1.5 g. primary standard potassium iodate dried at 180° C. and dilute to volume in a 500-ml. volumetric flask. To a 500-ml. iodine flask, pipet 50 ml. of iodate solution. Add 2 g. potassium iodide and 10 ml. of 1 N hydrochloric acid. Stopper the flask. After 5 minutes, titrate with stock thiosulfate solution to a pale yellow. Add 5 ml. starch indicator solution and continue the titration until the blue color disappears. Calculate the normality of the stock solution:

$$N = \frac{W}{M} \times 2.80$$

N = Normality of stock thiosulfate solution.
M = Volume of thiosulfate required, ml.
W = Weight of potassium iodate, grams.

6.2.7 *Sodium Thiosulfate Titrant (0.01 N).* Dilute 100 ml. of the stock thiosulfate solution to 1,000 ml. with freshly boiled distilled water.

Normality = Normality of stock solution × 0.100.

6.2.8 *Standardized Sulfite Solution for Preparation of Working Sulfite-TCM Solution.* Dissolve 0.3 g. sodium metabisulfite ($Na_2S_2O_5$) or 0.40 g. sodium sulfite (Na_2SO_3) in 500 ml. of recently boiled, cooled, distilled water. (Sulfite solution is unstable; it is therefore important to use water of the highest purity to minimize this instability.) This solution contains the equivalent of 320 to 400 µg./ml. of SO_2. The actual concentration of the solution is determined by adding excess iodine and back-titrating with standard sodium thiosulfate solution. To back-titrate, pipet 50 ml. of the 0.01 N iodine into each of two 500-ml. iodine flasks (A and B). To flask A (blank) add 25 ml. distilled water, and to flask B (sample) pipet 25 ml. sulfite solution. Stopper the flasks and allow to react for 5 minutes. Prepare the working sulfite-TCM Solution (6.2.9) at the same time iodine solution is added to the flasks. By means of a buret containing standardized 0.01 N thiosulfate, titrate each flask in turn to a pale yellow. Then add 5 ml. starch solution and continue the titration until the blue color disappears.

6.2.9 *Working Sulfite-TCM Solution.* Pipet accurately 2 ml. of the standard solution into a 100 ml volumetric flask and bring to mark with 0.04 M TCM. Calculate the concentration of sulfur dioxide in the working solution:

$$\mu g\ SO_2/ml. = \frac{(A - B)\ (N)\ (32{,}000)}{25} \times 0.02$$

A = Volume thiosulfate for blank, ml.
B = Volume thiosulfate for sample, ml.
N = Normality of thiosulfate titrant.
32,000 = Milliequivalent wt. of SO_2, µg.
25 = Volume standard sulfite solution, ml.
0.02 = Dilution factor.

This solution is stable for 30 days if kept at 5° C. (refrigerator). If not kept at 5° C., prepare daily.

6.2.10 *Purified Pararosaniline Stock Solution (0.2 percent nominal).*

6.2.10.1 *Dye Specifications.* The pararosaniline dye must meet the following performance specifications: (1) the dye must have a wavelength of maximum absorbance at 540 nm. when assayed in a buffered solution of 0.1 M sodium acetate-acetic acid; (2) the absorbance of the reagent blank, which is temperature-sensitive (0.015 absorbance unit/°C), should not exceed 0.170 absorbance unit at 22° C. with a 1-cm. optical path length, when the blank is prepared according to the prescribed analytical procedure and to the specified concentration of the dye; (3) the calibration curve (Section 8.2.1) should have a slope of 0.030±0.002 absorbance units/μg. SO_2 at this path length when the dye is pure and the sulfite solution is properly standardized.

6.2.10.2 *Preparation of Stock Solution.* A specially purified (99–100 percent pure) solution of pararosaniline, which meets the above specifications, is commercially available in the required 0.20 percent concentration (Harleco*). Alternatively, the dye may be purified, a stock solution prepared and then assayed according to the procedure of Scaringelli, et al. (4)

6.2.11 *Pararosaniline Reagent.* To a 250-ml. volumetric flask, add 20 ml. stock pararosaniline solution. Add an additional 0.2 ml. stock solution for each percent the stock assays below 100 percent. Then add 25 ml. 3 M phosphoric acid and dilute to volume with distilled water. This reagent is stable for at least 9 months.

7. *Procedure.*

7.1 *Sampling.* Procedures are described for short-term (30 minutes and 1 hour) and for long-term (24 hours) sampling. One can select different combinations of sampling rate and time to meet special needs. Sample volumes should be adjusted, so that linearity is maintained between absorbance and concentration over the dynamic range.

7.1.1 *30-Minute and 1-Hour Samplings.* Insert a midget impinger into the sampling system, Figure A1. Add 10 ml. TCM solution to the impinger. Collect sample at 1 liter/minute for 30 minutes, or at 0.5 liter/minute for 1 hour, using either a rotameter, as shown in Figure A1, or a critical orifice, as shown in Figure A1a, to control flow. Shield the absorbing reagent from direct sunlight during and after sampling by covering the impinger with aluminum foil, to prevent deterioration. Determine the volume of air sampled by multiplying the flow rate by the time in minutes and record the atmospheric pressure and temperature. Remove and stopper the impinger. If the sample must be stored for more than a day before analysis, keep it at 5° C. in a refrigerator (see 4.2).

7.1.2 *24-Hour Sampling.* Place 50 ml. TCM solution in a large absorber and collect the sample at 0.2 liter/minute for 24 hours from midnight to midnight. Make sure no entrainment of solution results with the impinger. During collection and storage protect from direct sunlight. Determine the total air volume by multiplying the air flow rate by the time in minutes. The correction of 24-hour measurements for temperature and pressure is extremely difficult and is not ordinarily done. However, the accuracy of the measurement will be improved if meaningful corrections can be applied. If storage is necessary, refrigerate at 5° C. (see 4.2).

7.2 *Analysis.*

7.2.1 *Sample Preparation.* After collection, if a precipitate is observed in the sample, remove it by centrifugation.

7.2.1.1 *30-Minute and 1-Hour Samples.* Transfer the sample quantitatively to a 25-ml. volumetric flask; use about 5 ml. distilled water for rinsing. Delay analyses for 20 minutes to allow any ozone to decompose.

7.2.1.2 *24-Hour Sample.* Dilute the entire sample to 50 ml. with absorbing solution. Pipet 5 ml. of the sample into a 25-ml. volumetric flask for chemical analyses. Bring volume to 10 ml. with absorbing reagent. Delay analyses for 20 minutes to allow any ozone to decompose.

7.2.2 *Determination.* For each set of determinations prepare a reagent blank by adding 10 ml. unexposed TCM solution to a 25-ml. volumetric flask. Prepare a control solution by adding 2 ml. of working sulfite-TCM solution and 8 ml. TCM solution to a 25-ml. volumetric flask. To each flask containing either sample, control solution, or reagent blank, add 1 ml. 0.6 percent sulfamic acid and allow to react 10 minutes to destroy the nitrite from oxides of nitrogen. Accurately pipet in 2 ml. 0.2 percent formaldehyde solution, then 5 ml. pararosaniline solution. Start a laboratory timer that has been set for 30 minutes. Bring all flasks to volume with freshly boiled and cooled distilled water and mix thoroughly. After 30 minutes and before 60 minutes, determine the absorbances of the sample (denote as A), reagent blank (denote as A_0) and the control solution at 548 nm. using 1-cm. optical path length cells. Use distilled water, not the reagent blank, as the reference. (NOTE! This is important because of the color sensitivity of the reagent blank to temperature changes which can be induced in the cell compartment of a spectrophotometer.) Do not allow the colored solution to stand in the absorbance cells, because a film of dye may be deposited. Clean cells with alcohol after use. If the temperature of the determinations does not differ by more than 2° C. from the calibration temperature (8.2), the reagent blank should be within 0.03 absorbance unit of the y-intercept of the calibration curve (8.2). If the reagent blank differs by more than 0.03 absorbance unit from that found in the calibration curve, prepare a new curve.

7.2.3 *Absorbance Range.* If the absorbance of the sample solution ranges between 1.0 and 2.0, the sample can be diluted 1:1 with a portion of the reagent blank and read within a few minutes. Solutions with higher absorbance can be diluted up to sixfold with

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*Hartmen-Leddon, 60th and Woodland Avenue, Philadelphia, PA 19143.

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the reagent blank in order to obtain onscale readings within 10 percent of the true absorbance value.

8. *Calibration and Efficiencies.*

8.1 *Flowmeters and Hypodermic Needle.* Calibrate flowmeters and hypodermic needle (*5*) against a calibrated wet test meter.

8.2 *Calibration Curves.*

8.2.1 *Procedure with Sulfite Solution.* Accurately pipet graduated amounts of the working sulfite-TCM solution (6.2.9) (such as 0, 0.5, 1, 2, 3, and 4 ml.) into a series of 25-ml. volumetric flasks. Add sufficient TCM solution to each flask to bring the volume to approximately 10 ml. Then add the remaining reagents as described in 7.2.2. For maximum precision use a constant-temperature bath. The temperature of calibration must be maintained within ±1° C. and in the range of 20° to 30° C. The temperature of calibration and the temperature of analysis must be within 2 degrees. Plot the absorbance against the total concentration in µg. SO_2 for the corresponding solution. The total µg. SO_2 in solution equals the concentration of the standard (Section 6.2.9) in µg. SO_2/ml. times the ml. sulfite solution added (µg. SO_2= µg./ml. SO_2×ml. added). A linear relationship should be obtained, and the y-intercept should be within 0.03 absorbance unit of the zero standard absorbance. For maximum precision determine the line of best fit using regression analysis by the method of least squares. Determine the slope of the line of best fit, calculate its reciprocal and denote as B_s. B_s is the calibration factor. (See Section 6.2.10.1 for specifications on the slope of the calibration curve). This calibration factor can be used for calculating results provided there are no radical changes in temperature or pH. At least one control sample containing a known concentration of SO_2 for each series of determinations, is recommended to insure the reliability of this factor.

8.2.2 *Procedure with SO_2 Permeation Tubes.*

8.2.2.1 *General Considerations.* Atmospheres containing accurately known amounts of sulfur dioxide at levels of interest can be prepared using permeation tubes. In the systems for generating these atmospheres, the permeation tube emits SO_2 gas at a known, low, constant rate, provided the temperature of the tube is held constant (±0.1° C.) and provided the tube has been accurately calibrated at the temperature of use. The SO_2 gas permeating from the tube is carried by a low flow of inert gas to a mixing chamber where it is accurately diluted with SO_2-free air to the level of interest and the sample taken. These systems are shown schematically in Figures A2 and A3 and have been described in detail by O'Keeffe and Ortman (*9*), Scaringelli, Frey, and Saltzman (*10*), and Scaringelli, O'Keeffe, Rosenberg, and Bell (*11*).

8.2.2.2 *Preparation of Standard Atmospheres.* Permeation tubes may be prepared or purchased. Scaringelli, O'Keeffe, Rosenberg, and Bell (*11*) give detailed, explicit directions for permeation tube calibration. Tubes with a certified permeation rate are available from the National Bureau of Standards. Tube permeation rates from 0.2 to 0.4 µg./minute, inert gas flows of about 50 ml./minute, and dilution air flow rates from 1.1 to 15 liters/minute conveniently give standard atmospheres containing desired levels of SO_2 (25 to 390 µg./m.^{3}; 0.01 to 0.15 p.p.m. SO_2). The concentration of SO_2 in any standard atmosphere can be calculated as follows:

$$C=\frac{P\times10^{3}}{R_d+R_i}$$

Where:

C = Concentration of SO_2, µg./m.^{3} at reference conditions.

P = Tube permeation rate, µg./minute.

R_d= Flow rate of dilution air, liter/minute at reference conditions.

R_i= Flow rate of inert gas, liter/minute at reference conditions.

8.2.2.3 *Sampling and Preparation of Calibration Curve.* Prepare a series (usually six) of standard atmospheres containing SO_2 levels from 25 to 390 µg. SO_2/m.^{3}. Sample each atmosphere using similar apparatus and taking exactly the same air volume as will be done in atmospheric sampling. Determine absorbances as directed in 7.2. Plot the concentration of SO_2 in µg./m.^{3} (x-axis) against $A-A_0$ values (y-axis), draw the straight line of best fit and determine the slope. Alternatively, regression analysis by the method of least squares may be used to calculate the slope. Calculate the reciprocal of the slope and denote as B_g.

8.3 *Sampling Efficiency.* Collection efficiency is above 98 percent; efficiency may fall off, however, at concentrations below 25 µg./m.^{3}. (*12, 13*)

9. *Calculations.*

9.1 *Conversion of Volume.* Convert the volume of air sampled to the volume at reference conditions of 25° C. and 760 mm. Hg. (On 24-hour samples, this may not be possible.)

$$V_R=V\times\frac{P}{760}\times\frac{298}{t+273}$$

V_R= Volume of air at 25° C. and 760 mm. Hg, liters.

V = Volume of air sampled, liters.

P = Barometric pressure, mm. Hg.

t = Temperature of air sample, °C.

9.2 *Sulfur Dioxide Concentration.*

9.2.1 When sulfite solutions are used to prepare calibration curves, compute the concentration of sulfur dioxide in the sample:

$$\mu g.\ SO_2/m.^3 = \frac{(A-A_o)\ (10^3)\ (B_s)}{V_R} \times D$$

A = Sample absorbance.
A_o = Reagent blank absorbance.
10^3 = Conversion of liters to cubic meters.
V_R = The sample corrected to 25° C. and 760 mm. Hg, liters.
B_s = Calibration factor, μg./absorbance unit.
D = Dilution factor.
For 30-minute and 1-hour samples, $D = 1$.
For 24-hour samples, $D = 10$.

9.2.2 When SO_2 gas standard atmospheres are used to prepare calibration curves, compute the sulfur dioxide in the sample by the following formula:

$$SO_2,\ \mu g./m.^3 = (A - A_o) \times B_g$$

A = Sample absorbance.
A_o = Reagent blank absorbance.
B_g = (See 8.2.2.3).

9.2.3 *Conversion of μg./m.³ to p.p.m.*—If desired, the concentration of sulfur dioxide may be calculated as p.p.m. SO_2 at reference conditions as follows:

$$p.p.m.\ SO_2 = \mu g.\ SO_2/m.^3 \times 3.82 \times 10^{-4}$$

10. *References.*

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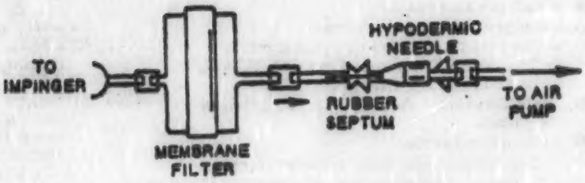
TO IMPINGER
HYPODERMIC NEEDLE
RUBBER SEPTUM
TO AIR PUMP
MEMBRANE FILTER

Figure A1a. Critical orifice flow control.

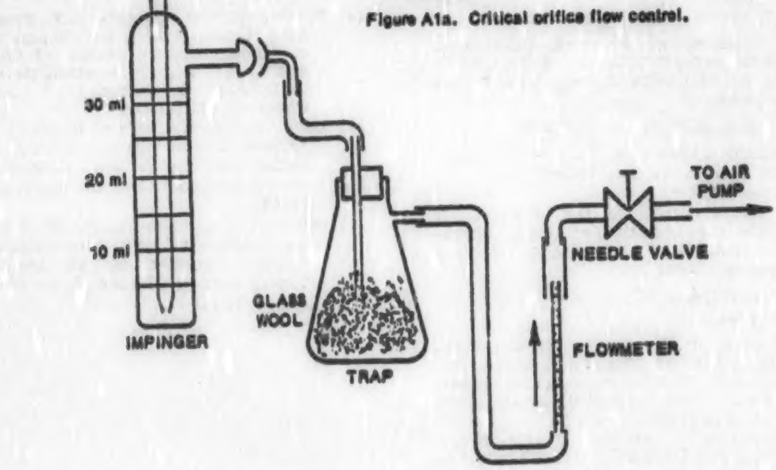
30 ml
20 ml
10 ml
IMPINGER
GLASS WOOL
TRAP
TO AIR PUMP
NEEDLE VALVE
FLOWMETER

Figure A1. Sampling train.

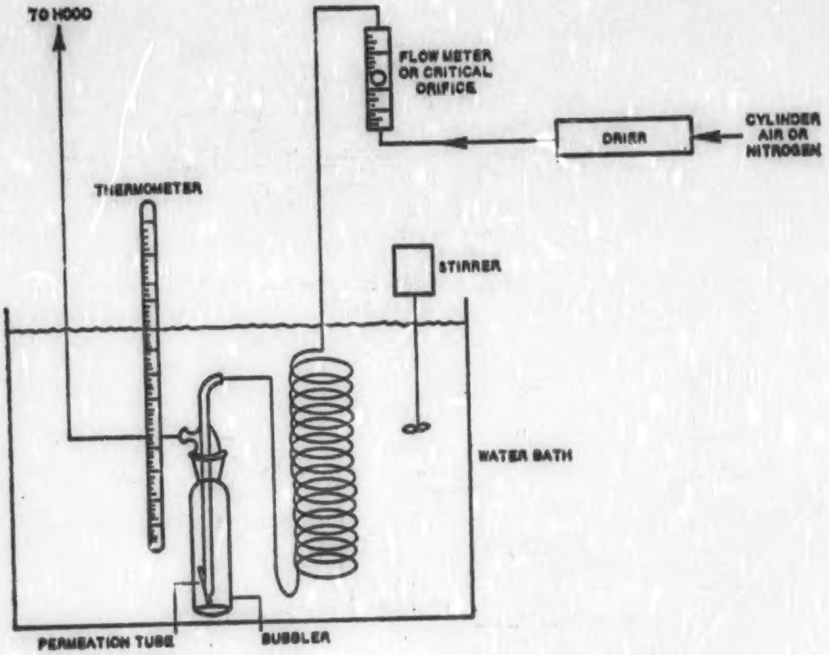
TO HOOD
FLOW METER OR CRITICAL ORIFICE
DRIER
CYLINDER AIR OR NITROGEN
THERMOMETER
STIRRER
WATER BATH
PERMEATION TUBE
BUBBLER

Figure A2. Apparatus for gravimetric calibration and field use.

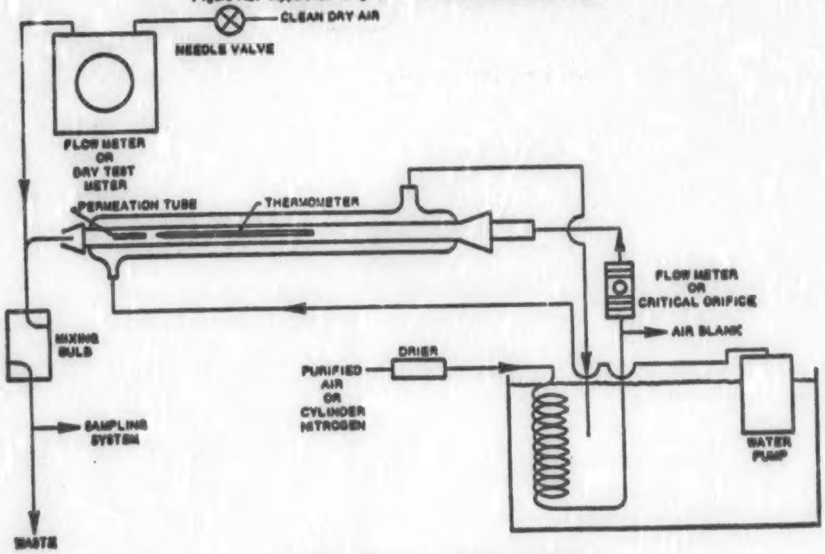
CLEAN DRY AIR
NEEDLE VALVE
FLOW METER OR DRY TEST METER
PERMEATION TUBE
THERMOMETER
FLOW METER OR CRITICAL ORIFICE
AIR BLANK
MIXING BULB
DRIER
PURIFIED AIR OR CYLINDER NITROGEN
WATER PUMP
SAMPLING SYSTEM
WASTE

Figure A3. Permeation tube schematic for laboratory use.

11.0 REFERENCES

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12.0 DATA FORMS

Blank data forms are provided on the following pages for the convenience of the Handbook user. Many of these forms are new, while others have been adapted from EPA forms and those of Reference 1. No documentation is given on these forms as it would detract from their usefulness. The titles are also placed at the top of the figure as is customary for a data form. In order to relate the form to the test method, a form number is given in the lower right-hand corner, e.g.,

Form SO_2-2.17,

which implies that the form is Figure 2.17 in Section 2 of the SO_2 Handbook. Future revisions of these forms, if any, can be documented by 2.17A, 2.17B, etc. The data forms included are listed below.

Form	Title
1.1	Procurement Log
2.2	Wet Test Meter Calibration Log
2.4	Soap Bubble Meter Calibration Log
2.6	Mass Flow Meter Calibration Log
2.8	Rotameter Calibration Data Sheet for SO_2 Sampling Train
2.12	Calibration Log for Critical Flow Orifices
2.17	Timer Calibration Log
4.9	Gas Bubbler Data Record
5.1A	Laboratory Data Log (Front)
5.1B	Laboratory Data Log (Back)
6.1	SAROAD Daily Data Form
6.2	SAROAD Hourly Data Form
8.1	$\overline{X}$ and R Chart

PROCUREMENT LOG

Item description	Qty	Purchase order No.	Vendor	Date		Cost	Disposition	Comments
				Ord	Rec			

WET TEST METER CALIBRATION LOG

Wet Test Meter Serial No. ____________ Date ____________

Wet Test Meter Range ______ Calibrated by ____________ Volume of Test Flask, V_{st} = ______

Test No.	Wet Test Meter Temperature		Test Data				Wet Test Meter Readings, ℓ			V_s* ℓ	Remarks
			Barometric Pressure, P_B	Manometer Reading, Δp		Barometric Pressure Corrected $P_B - \Delta p$					
	°C	T_m in °K	mm Hg	mm H_2O	mm Hg	P_m in mm Hg	V_f	V_i	V_m		

Calculations:

T_m = _____ °C + 273.16 = ______ °K

Δp(mm Hg) = 0.0738 Δp(mm H_2O) = 0.0738 x ____ = ____ mm Hg

$P_m = P_B - \Delta p$ = _____ - _____ = ______ mm Hg

T_s = 298.16°K

P_s = 760 mm Hg

$V_m = V_f - V_i$ = ______ ℓ

*$V_s = P_m/P_s \times T_s/T_m \times V_m$ = ______ ℓ

$\overline{V}_s$ = Average of V_s values for the three tests = ______ ℓ

% Error = 100 $(\overline{V}_s - V_{st})/V_{st}$ = ______ %, not to exceed ±1%

SOAP BUBBLE METER CALIBRATION LOG

Meter Serial No.____________________ Date____________________________

Displacement Volume_________________Calibrated by__________________

Test No.	Water Temperature °C	Wt_i g	Wt_f g	V ml	Remarks

Calculations:

D_r = ____g/ml, from Table 2.1

$$V_d = \frac{Wt_f - Wt_i}{D_r}$$

$$\bar{V}_d = (V_{d_1} + V_{d_2} + V_{d_3})/3 = \_\_\_\_\_\_\_\_\_\_$$

V = Displacement volume stated by manufacturer = _____ml

$$\% \text{ Error} = \frac{(\bar{V}_d - V) \times 100}{V} = \_\_\_\_\_\_\_\_\_\_$$, not to exceed 1%.

MASS FLOW METER CALIBRATION LOG

Mass Flow Meter Serial No.____________________ Calibrated with________________

Location____________________ Ambient/Room Temperature________________ ^{o}K

Barometric Pressure (P_B)____________________ Relative Humidity________________

Calibrated by____________________ Date________________

mm Hg = 0.0738 × mm H_2O

Test Point	Wet Test Meter Readings				$V_s = \frac{P_m T_s}{P_s T_m} V_m$	Elapsed Time t (min)	$Q_s = \frac{V_s}{t}$ (ℓ/min)	Mass Flow Meter Scale
	Manometer Reading Δp (mm Hg)	$P_m = P_B - \Delta p$ P_m (mm Hg)	Meter Fluid Temperature T_m (^{o}K)	Volume Displaced V_m (ℓ)				
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								

ROTAMETER CALIBRATION DATA SHEET FOR SO_2 SAMPLING TRAIN

Rotameter Serial No.__________ Calibrated with__________

Location__________ Ambient/Room Temperature______ $^oC + 273.16 =$ ___ ^oK

Barometric Pressure (P_B)__________ Relative Humidity__________

Calibrated by__________ Date__________

Test Point	Rotameter Reading units	Elapsed Time t (min)	Temperature Liquid C^o	Manometer Reading mm H_2O	Volume Displaced V_m (ℓ)	$V_s = \frac{P_m T_s}{P_s T_m} V_m$	$Q_s = \frac{V_s}{t}$ (ℓ/min)	Remarks
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								

CALIBRATION LOG FOR CRITICAL FLOW ORIFICES

Needle Identification Number	Date of Calibration	Calibration Method	Flow Rate at STP*	Analyst	Remarks

TIMER CALIBRATION LOG

Site location	Timer No.	Date		Elapsed time indicator serial No.	Test period	Elapsed time indicator reading	Accuracy	By (signature)
		Start	Stop					

GAS BUBBLER DATA RECORD

		OPEN	CLAMP
STATION LOCATION	VACUUM READING START	___	___
CITY & STATE ____________________	VACUUM READING END	___	___

SITE ADDRESS ____________________

START SAMPLING ___ ___ ___ ___ ___
mo day yr hr min

PROJECT __________ SITE NO. __________

STOP SAMPLING ___ ___ ___ ___ ___
mo day yr hr min

POLLUTANT ____________________

SAMPLER ID NO. ____ SAMPLE NO. __________

REMARKS ____________________
____
____

WIND: ____calm, ___light, ___gusty
VISIBILITY: ___clear, ___hazy
SKY: ___clear, ___scattered, ___overcast
HUMIDITY: ___dry, ___moderate, ___humid, ___rain
TEMP. °F: ___<20, ___20-40, ___41-60, ___61-80,
___>80

These meteorological conditions are for use on those occasions when an anomally occurs.

Sample was collected in accordance with the guidelines set forth on the reverse side of this card.

____
Signature

Front of card

-Proper flow rate-rotameter - ½ hr samples - 900-1100 cm³/min.
1 hr samples - 450-550 cm³/min.
24 hr samples - 180-220 cm³/min.

- critical orifice calibrated prior to use
- flow meter should be calibrated after 30 days of operation or at least every 6 months.

-24-hour samples

- 23 hrs < sampling time < 25 hrs
- At least 35 ml of absorbing reagent must remain after sampling
- Sampler timer accuracy is ±15 min. per 24 hrs.

Back of card

Quality Assurance Handbook SO_2-4.9

LABORATORY DATA LOG (Front)

GAS BUBBLER - SO_2

NAME PARAMETER CODE 4 2 4 0 1

CITY NAME ____________ STATE ☐☐ AREA ☐☐☐☐ SITE ☐☐☐

SITE ADDRESS ____________ PROJECT ☐☐ TIME ☐ YEAR ☐☐

PROJECT ____________ TIME INTERVAL

SAMPLER IDENTIFICATION NO.____________

SAMPLING DATE			CONC	SAMPLE	FLOW RATE ft^3/min			SAMPLING AIR m^3		OPTICAL	B_s	CONC. of	REMARKS
MONTH	DAY	ST HR	$\mu g/m^3$	NUMBER	START	END	AVERAGE	TIME	VOLUME	DENSITY	µg/OD unit	SOL'N.	

When sulfite solutions are used to prepare calibration curves, compute the concentration of sulfur dioxide in the sample:

$$\mu g\ SO_2/m^3 = \frac{(A-A_0)\ (10^3)\ (B_s)}{V_R} \times D$$

A = Sample absorbance.

A_0 = Reagent blank absorbance

10^3 = Conversion of liters to cubic meters.

V_R = The sample corrected to 25° C. and 760mm. Hg, liters.

B_s = Calibration factor, µg./absorbance unit.

D = Dilution factor.

For 30-minute and 1-hour samples, D = 1.

For 24-hour samples, D = 10.

ALL SAMPLES WERE HANDLED IN ACCORDANCE WITH THE GUIDE LINES SET FORTH ON THE REVERSE SIDE OF THIS CARD

____
SIGNATURE

LABORATORY DATA LOG (Back)

PREPARATION AND CALIBRATION

Reagent water must be double distilled and be stored in a borosilicate glass vessel, capped and with a vent guard.

The absorbing reagent must be prepared every 6 months, be clear and have 3.0 pH < 5.0.

24-hour samples were polypropylene tube and closure with glass impinger 1/2- and 1-hour samples use all glass midget impinger.

Critical orifice should be calibrated with rotameter (24-hr sampling) or soap bubble meter or wet test meter (1/2- and 1-hr sampling) or mass flow meter.

All data points within ±2% of the best-fit calibration curve.

Critical orifice should be calibrated before and after each sampling period.

Rotameter cleaned and calibrated every 30 days of operation or every 6 months.

Sample timer should be checked to be accurate within ±15 min/24-hr. every 6 months.

SAMPLE ANALYSIS

Colorimetric analysis-Blank within ±0.03 absorbance unit of absorbance calibration intercept. Control sample absorbance within ±1.2 μg SO_2 of actual value.

Calibration curve for absorbance vs. μg SO_2/m^3 slope between 0.03 ± 0.002 absorbance units/μg SO_2. Intercept below 0.17. All points within 0.8 μg SO_2 of best-fit curve.

Reference samples should be within ±1.2 μg SO_2 of correct value. Any 2 consecutive results outside ±0.8 μg SO_2 in the same direction are unacceptable.

Reproduced from best available copy.

REAGENT PREPARATION

Reagent	Container	Frequency of preparation
Sulfamic acid, 0.6%	Glass stoppered	Prior to analysis
Formaldehyde, 0.2%	Glass stoppered	Prior to analysis
Working iodine solution, 0.01 n	Glass stoppered, dark	Prior to analysis
Stock iodine solution, 0.1 n	Glass stoppered, dark	Yearly
Sodium thiosulfate titrant, 0.01 n	Glass stoppered	Yearly
Stock sodium thiosulfate, 0.1 n	Glass stoppered	Yearly

Working sulfite TCM solution should be accurately prepared from standard solution prior to an analysis and is stable for 30 days @ 5°C when stored in a dark glass stoppered bottle.

Standard sulfite TCM solution 320-400 μg/ml SO_2.

Pararosaniline reagent 100% assay is stable when stored in a dark glass stoppered bottle.

Assayed stock pararosaniline solution should be stored in a dark glass stoppered bottle. Prepare new solution when the slope of the calibration curve deviates from 0.03 ± 0.002.

Analytical balance performance should be within ±0.1 mg of manufacturer's specification.

SAROAD DAILY DATA FORM

24-HOUR OR GREATER SAMPLING INTERVAL

2
1

Agency

City Name

Site Address

Project

Time Interval

State			Area				Site	
2	3	4	5	6	7	8	9	10

Agency	Project	Time	Year	Month
11	12 13	14	15 16	17 18

Name PARAMETER Code	Name PARAMETER Code	Name PARAMETER Code	Name PARAMETER Code
23 24 25 26 27	37 38 39 40 41	51 52 53 54 55	65 66 67 68 69
Method 28 29	Method 42 43	Method 56 57	Method 70 71
Units 30 31	Units 44 45	Units 58 59	Units 72 73
DP 32	DP 46	DP 60	DP 74

Day	St Hr				
19 20	21 22	33 34 35 36	47 48 49 50	61 62 63 64	75 76 77 78
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
26					
27					
28					
29					
30					
31					
	DP →	4 1 2 1 0	4 1 2 1 0	4 1 2 1 0	4 1 2 1 0

SAROAD HOURLY DATA FORM

LESS THAN 24-HOUR SAMPLING INTERVAL

☐ 1

1

Agency

City Name

Site Address

Project

ENVIRONMENTAL PROTECTION AGENCY
National Aerometric Data Bank
P. O. Box 12055
Research Triangle Park
North Carolina 27711

Parameter observed

Method

Time interval of obs.

Units of obs.

State		Area				Site		
☐	☐	☐	☐	☐	☐	☐	☐	☐
2	3	4	5	6	7	8	9	10

Agency	Project		Time	Year		Month	
☐	☐	☐	☐	☐	☐	☐	☐
11	12	13	14	15	16	17	18

Parameter code					Method		Units		DP
☐	☐	☐	☐	☐	☐	☐	☐	☐	☐
23	24	25	26	27	28	29	30	31	32

Day	St Hr	Project Rdg 1	Rdg 2	Rdg 3	Rdg 4	Rdg 5	Rdg 6	Rdg 7	Rdg 8	Rdg 9	Rdg 10	Rdg 11	Rdg 12
19 20	21 22	33 34 35 36	37 38 39 40	41 42 43 44	45 46 47 48	49 50 51 52	53 54 55 56	57 58 59 60	61 62 63 64	65 66 67 68	69 70 71 72	73 74 75 76	77 78 79 80

$\overline{X}$ AND R CHART

PROJECT NAME ____________________ MEASUREMENT PERFORMED ____________________ MEASUREMENT UNITS ____________

DATE																											
MEASUREMENT	CODE	1																									
		2																									
		3																									
	RESULT	1																									
		2																									
		3																									
SUM																											
AVERAGE, $\overline{X}$																											
RANGE, R																											
			1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25
AVERAGES, $\overline{X}$																											
RANGES, R																											
Comments (Correct. Action, etc.)																											

SECTION 2.2

REFERENCE METHOD FOR THE DETERMINATION OF SUSPENDED PARTICULATES IN THE ATMOSPHERE (HIGH VOLUME METHOD)

OUTLINE

SUMMARY

METHOD DESCRIPTION

Section	Documentation	Number of Pages
1. PROCUREMENT OF EQUIPMENT AND SUPPLIES	2.2.1	3
2. CALIBRATION OF EQUIPMENT	2.2.2	25
3. FILTER SELECTION AND PREPARATION	2.2.3	5
4. SAMPLING PROCEDURE	2.2.4	17
5. ANALYSIS OF SAMPLES	2.2.5	3
6. CALCULATIONS AND DATA REPORTING	2.2.6	3
7. MAINTENANCE	2.2.7	5
8. AUDITING PROCEDURE	2.2.8	12
9. RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY	2.2.9	1
10. REFERENCE METHOD	2.2.10	5
11. REFERENCES	2.2.11	1
12. DATA FORMS	2.2.12	8

SUMMARY

Air is drawn into a covered housing and through a filter by means of a high-flow-rate blower at a flow rate (1.13 to 1.70 m^3/min; 40 to 60 ft^3/min) that allows suspended particles having diameters of less than 100 μm (Stokes equivalent diameter) to collect on the filter surface. Particles within the size range of 100 to 0.1 μm diameter are ordinarily collected on glass fiber filters. The mass concentration of suspended particulates in the ambient air (μg/m^3) is computed by measuring the mass of collected particulates and the volume of air sampled.

This method is applicable to measurement of the mass concentration of suspended particulates in ambient air. The size of the sample collected is usually adequate for other analyses.

When the sampler is operated at an average flow rate of 1.70 m^3/min (60 ft^3/min) for 24 hours, an adequate sample will be obtained even in an atmosphere having concentrations of suspended particulates as low as 1 μg/m^3.

Based on more extensive review and on the experience gained in collaborative tests[1] and in system audits, the method description and the limits recommended in the quality checks and audits, herein, are in some cases different from and supercede those in the guideline document.[2] On the other hand, Reference 2 contains functional and error analyses that are not included in this Handbook.

Based upon collaborative testing, the relative standard deviation for single analyst variation (repeatability of the method) is 3.9 percent. The corresponding value for multilaboratory variation (reproducibility of the method) is 5.6 percent.[1]

The method description which follows is based on the promulgated reference method published in the Code of Federal Regulations 40, Part 50.11, Appendix B, July 1, 1975, pages 12-16. A complete copy of the reference method is contained in Section 10.

METHOD DESCRIPTION

1.0 PROCUREMENT OF EQUIPMENT AND SUPPLIES

Specifications for the apparatus and supplies for monitoring ambient air for total suspended particulates (TSP) are presented in the Reference Method, Section 10.

Upon receipt of the sampling equipment, apparatus and supplies, procurement checks should be conducted (some of these checks are presented in Table 1.1, Activity Matrix for Procurement of Equipment and Supplies). These checks should be recorded in a procurement log. See Figure 1.1 for an example and Section 2.2.12 (Data Forms) for a blank copy for use by the Handbook user. The procurement log will serve as a permanent record for future procurements. This log will provide continuity of equipment and supplies and will also provide records for future program fiscal projections. The following tabulation of equipment, apparatus and supplies is presented with the reference to subsections within this section as a guide to the user for finding specific information as to the check out procedures that are presented in this Handbook.

Item	Reference to subsections within this section
Analytical balance	2.1
Relative humidity indicator	2.2
Elapsed time meter	2.3
Timer	2.4
Orifice calibration unit	2.5
Sampler	2.6
Filter	1-3.3
Sampler motor	7.1
Faceplate gasket	7.2
Rotameter	7.3
Sampling head	7.4
Motor gasket	7.5
Flow transducer and recorder	7.6

Item description	Qty	Purchase order No.	Vendor	Date		Cost	Disposition	Comments
				Ord	Rec			
Hi-Vol Brush Set	12	1782	Gen'l Metal	6/1/75	6/10/75	$21	Acc.	
Hi Vol Filters	100	1782	" "	"	"	$25	Acc.	

Figure 1.1 Example of a procurement log

Table 1.1 ACTIVITY MATRIX FOR PROCUREMENT OF EQUIPMENT AND SUPPLIES

Equipment	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Analytical balance	Indicated weight = standard weight ± 0.0005 grams for 3 to 5 standard weights over sample-filter weight range.	On receipt check against weights of known accuracy	Request a recalibration by manufacturer/supplier
Elapsed time meter	24 hrs ± 2 minutes	On receipt check against standard timepiece of known accuracy	Reject or adjust
Orifice calibration unit	Flow rate from manufacturer = actual flow rate ± 4%	On receipt check against flow rate primary standard	1) Adopt new calibration curve if evidence of damage is absent 2) Reject if evidence of damage is present
Sampler	Sampler complete Absence of evidence damage	Visual observation	Reject or repair
Relative humidity indicator	Indicator reading = psychrometer reading ± 6%.	On receipt compare with reading of wet bulb-dry bulb psychrometer	Adjust or replace to attain acceptance limits

2.0 CALIBRATION OF EQUIPMENT

Prior to the implementation of a sampling program for TSP, a wide spectrum of sampling and analysis equipment must be calibrated. The calibration activities are summarized in Table 2.1, Activity Matrix for Calibration of Equipment, located at the end of this section. Many of these calibration checks should also be employed for the initial procurement check activities. All data and calculations involved in these calibration activities should be recorded in a separate calibration log book. It is suggested that this log be arranged such that a separate section is designated for each apparatus and sampler employed in the program.

2.1 Analytical Balance

The balance calibration should be verified when the balance is 1) first purchased, 2) any time the balance has been moved or subjected to rough handling, or 3) during routine operations when a standard weight cannot be weighed within ± 0.5 mg of its stated weight. Covering the weight range normally encountered in weighing filters, weigh a set of three to five standard weights. An example of a typical balance performance log is presented in Figure 2.1. If at any time one or more of the standard weights cannot be measured within ± 0.5 mg of its stated value, have the balance recalibrated. The manufacturer should perform the calibration and subsequent adjustments. Record the results of all balance checks in the log book, see Figure 2.1.

2.2 Relative Humidity Indicator

The relative humidity indicator used for monitoring the conditioning environment should be checked against a wet-bulb, dry-bulb psychrometer or equivalent every six months. At least a two-point calibration should be made by comparing readings made in the conditioning environment and then moving the relative humidity indicator outdoors or perhaps just out of the conditioning room for a second comparison. If the

HIGH VOLUME FILTER WEIGHING QUALITY CONTROL LOG

Date	Time	Class "S" Weights (g)			Technician
		0.5000	1.0000	2.0000	
7/29/74	11:07	0.5000	1.0002	2.0000	BSM
7/29/74	12:08	0.5002	1.0003	2.0001	DEK
7/29/74	2:40	0.5000	1.0000	1.9999	DEK
7/30/74	4:03	0.4996	0.9999	2.0002	JLK
7/31/74	9:57	0.4994	1.0000	2.0000	DEK
7/31/74	10:56	0.4995	0.9992	1.9993	DEK
7/31/74	11:57	0.4994	0.9994	1.9994	DEK
7/31/74	2:04	0.5001	1.0000	2.0002	DEK
7/31/74	3:05	0.5000	1.0000	2.0000	DEK
8/1/74	9:03	0.4998	0.9992	1.9992	DEK
8/1/74	10:05	0.4993	0.9992	1.9992	DEK
8/1/74	11:10	0.5000	1.0001	2.0001	DEK
8/1/74	12:12	0.4998	0.9997	1.9993	DEK
8/1/74	1:43	0.5000	1.0001	2.0002	DEK
8/1/74	2:42	0.5001	1.0001	2.0001	DEK
8/1/74	3:45	0.5001	1.0000	2.0001	DEK
8/2/74	8:54	0.5000	1.0001	2.0001	DEK
8/2/74	9:56	0.5000	1.0000	2.0001	DEK
8/2/74	10:59	0.5003	0.9999	1.9998	DEK
8/2/74	12:16	0.5001	1.0002	2.0002	DEK
8/2/74	1:55	0.4990	1.0002	2.0001	DEK
8/2/74	3:03	0.5000	0.9999	2.0001	DEK
8/2/74	4:00	0.5000	0.9998	1.9999	DEK
8/3/74	8:41	0.4999	0.9996	1.9998	DEK
8/3/74	11:15	0.5002	1.0002	2.0002	DEK
8/5/74	8:42	0.5001	1.0000	2.0000	DEK
8/5/74	9:45	0.5000	1.0000	2.0000	DEK
8/5/74	10:44	0.5000	1.0000	2.0000	DEK
8/5/74	11:46	0.5000	1.0001	2.0000	DEK
8/5/74	1:16	0.5001	1.0000	2.0000	DEK
8/5/74	2:21	0.5001	1.0000	2.0001	DEK
8/5/74	3:15	0.5000	1.0000	2.0001	DEK
8/6/74	9:37	0.4999	1.0000	2.0000	DEK
8/6/74	11:05	0.5000	0.9998	1.9994	DEK
8/6/74	12:10	0.4999	0.9998	1.9999	DEK
8/6/74	2:10	0.5000	0.9999	2.0000	DEK
8/6/74	3:09	0.5000	1.0000	2.0000	DEK
8/6/74	4:05	0.5000	1.0000	2.0000	DEK
8/7/74	8:50	0.5000	1.0002	2.0003	DEK
8/7/74	9:46	0.4996	0.9992	1.9990	DEK
8/7/74	1:10	0.5001	1.0000	2.0000	DEK
8/7/74	2:20	0.5001	1.0000	2.0000	DEK
8/7/74	3:25	0.5002	1.0001	2.0000	DEK
8/8/74	9:46	0.5000	1.0000	2.0000	DEK
9/24/74	3:50	0.5001	1.0001	2.0001	DEK
9/26/74	3:01	0.5001	1.0001	2.0001	DEK

Figure 2.1 Example of balance performance record

difference between the indicator and corresponding psychrometer readings are within ±6 percent continue to use the relative humidity indicator. If they disagree by more than ±6 percent, either have the indicator calibrated or purchase a new one.

Record results of the relative humidity indicator checks in the calibration log book.

2.3 Elapsed Time Meter

The elapsed time meter (synchronous motor type 60 hertz) should be checked on site or in the laboratory every six months against a timepiece of known accuracy. If the indicator shows any signs of being temperature sensitive, it should be checked on site during each season of the year.

A gain or loss of more than 2 minutes in a 24-hour period warrants an adjustment or replacement of the indicator.

Record results of these checks in the calibration log book.

2.4 Timer

For those samplers that are equipped with an on-off timer, the timer should be calibrated and adjusted using a calibrated elapsed time meter as the reference. An example of this type calibration procedure is presented below. Figure 2.2 depicts the wiring diagram for use in this calibration.

The timer calibration procedure should be performed on a quarterly basis. Calibration data are recorded in the Timer Calibration Log. See Figure 2.3 for an example and Section 2.2.12 (Data Forms) for a blank copy for use by the Handbook user. The steps in the calibration procedure are:

1. Plug a correctly wired timer into an electrical outlet.
2. Set the timer to the correct time.
3. Set the ON and OFF time trippers for a 24-hour test period.
4. Plug the test light into one of the output plugs and an elapsed time meter into the other.
5. Check the system by manually operating the switch ON and OFF.

6. Allow the system to operate for the 24-hour test period and determine the elapsed time from the elapsed time meter.

   a. If the elapsed time is 24 hours ± 15 minutes, the timer is acceptable for field use.
   b. If the elapsed time is not 24 hours ± 15 minutes, adjust the tripper switches and repeat the test.

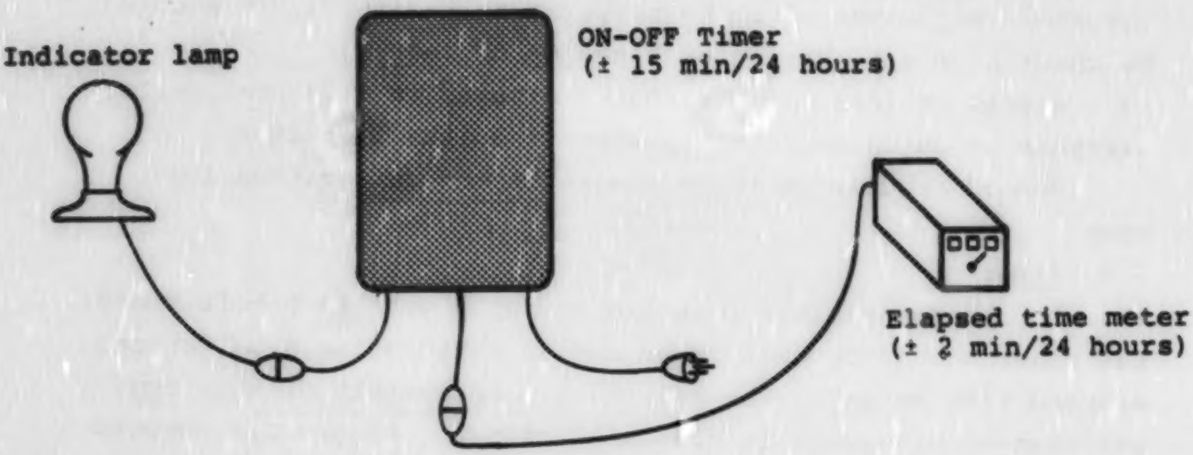


Figure 2.2 Diagram of a timer calibration system

2.5 Orifice Calibration Unit

The orifice calibration unit should be calibrated against a secondary standard, for example a Rootsmeter, upon receipt and at one-year intervals thereafter. The manufacturer's average calibration curve can be used unless the calibration deviates from it by more than ± 4 percent at any one point along the curve. When deviations from the manufacturer's curve are larger than ± 4 percent and there are no visible signs of damage to the orifice, the calibration should be repeated by another operator. If the large deviations persist (after the secondary standard has been checked

Site location	Timer No.	Date		Elapsed time indicator serial No.	Test period	Elapsed time indicator reading	Accuracy	By (signature)
		Start	Stop					
1141	4321	6/10/75	6/11/75	CW080G	24 hrs.	23 hrs. 53 min	99.5	C. Jones

Figure 2.3 Example of a timer calibration log

and found satisfactory) a new average calibration curve is constructed using the results from at least five sets of calibration data.

Orifice units should be visually inspected for visible signs of damage to the orifice before each use. A calibration check should be made if the orifice appears to have any nicks or dents.

The following stepwise orifice calibration procedure is adapted from Reference 3.

1. Assemble the parts as shown in Figure 2.4.
2. Zero the water and mercury manometers by sliding their scales until the zero on the scale is level with the meniscus as illustrated in Figure 2.5.
3. Check the level of the positive displacement meter table. Adjust the legs if necessary.
4. Install Load Plate 18 between the orifice and the positive displacement meter.
5. Turn Hi Vol motor ON, and let the system operate 5 minutes. While the unit equilibrates, continue with steps 6-9 below.
6. Write Plate #18 under the Plate # Column in the Hi Vol Orifice Calibration Log. See Figure 2.6 for an example and Section 2.2.12 (Data Forms) for a blank copy for use by the Handbook user.
7. Record the date, time, orifice number, name of primary standard (positive displacement meter) and the serial number of the primary standard in the appropriate spaces in the log.
8. Record the temperature in °C.

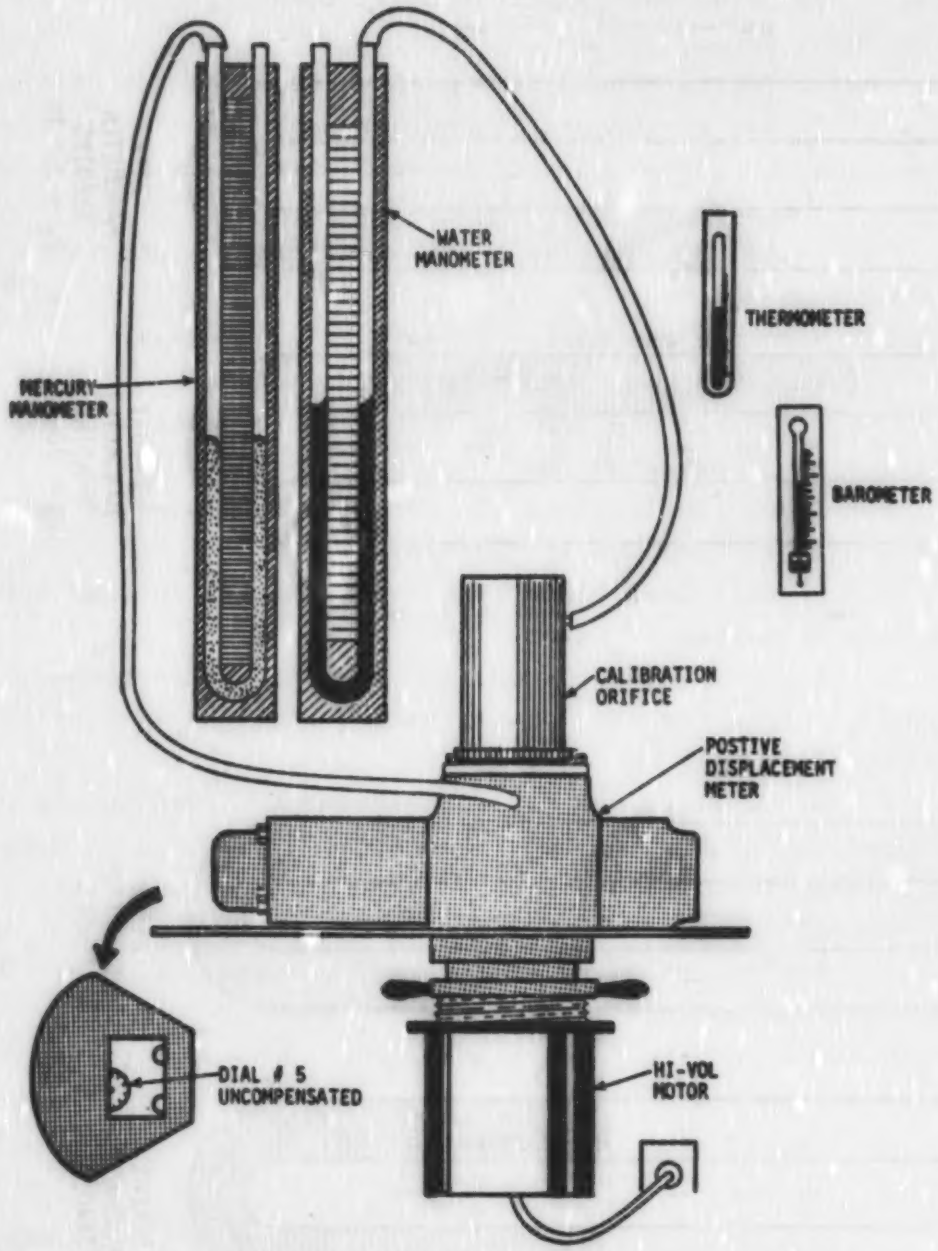

WATER MANOMETER
THERMOMETER
MERCURY MANOMETER
BAROMETER
CALIBRATION ORIFICE
POSTIVE DISPLACEMENT METER
DIAL # 5 UNCOMPENSATED
HI-VOL MOTOR

Figure 2.4 Diagram of positive displacement meter system

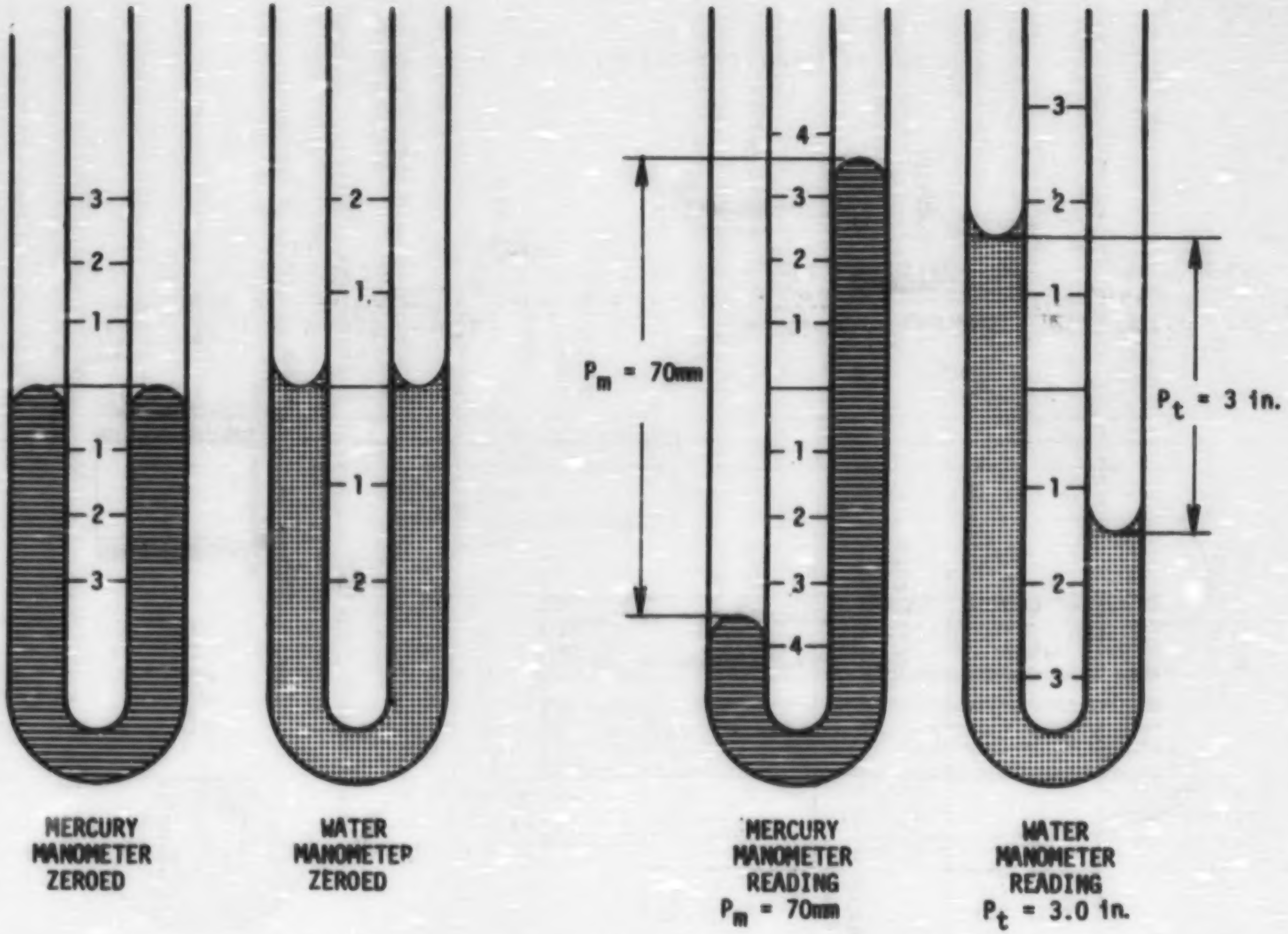

3
2
1
1
2
3
MERCURY
MANOMETER
ZEROED
2
1
1
2
WATER
MANOMETEP
ZEROED
P_m = 70mm
4
3
2
1
1
2
3
4
MERCURY
MANOMETER
READING
P_m = 70mm
3
2
1
P_t = 3 in.
1
2
3
WATER
MANOMETER
READING
P_t = 3.0 in.

Figure 2.5 How to read mercury and water manometers

Description Derivation	Ft.^{3} of Air	m^3 of Air	Barometric Pressure mm Hg	Vacuum in Standard in mm Hg	Absolute volume in m^3 $V_m \frac{(P_a - P_m)}{P_a}$	Time in Minutes	Flow rate m^3/min $\frac{V_a}{t}$	Pressure Drop Across the Orifice in inches of water
Symbol / Plate Number	V_c	V_m	P_a	P_m	V_a	t	Q_1	P_t
5	100	2.83	767	70	2.57	2.73	0.94	2.5
7	100	2.83	767	70	2.57	1.84	1.4	5.6
13	100	2.83	767	70	2.57	1.43	1.8	11.0
15	100	2.83	767	70	2.57	1.29	2.0	16.5
18	100	2.83	767	70	2.57	1.22	2.1	18.3
	100	2.83						
	100	2.83						
	100	2.83						
	100	2.83						
	100	2.83						

Orifice Number 4861
Manufacturer General Metal Works, Inc.
Temperature 26.0 °C
Barometric Pressure 767
Time 10:45 a.m. EST
Date 6-2-75
Primary Standard Roots Meter
Serial Number 10429
Calibration voltage 130
Signed J.J. Smith
Verified By John Young

Figure 2.6 Example of hi vol orifice calibration log

Definitions for use with Figure 2.6

V_a = Actual volume of air measured in cubic meters.

V_c = Volume measured by the positive displacement meter in cubic feet

V_m = Volume measured by the positive displacement meter in cubic meters as calculated from Vc.

P_a = Atmospheric pressure in mm Hg.

P_m = Vacuum at the inlet of the positive displacement meter in mm Hg.

t = Minutes of time elapsed during run.

Q_1 = Flow rate in cubic meters per minute at prevailing atmospheric pressure and temperature (uncorrected).

P_t = Pressure drop across the orifice in inches of water.

Equations given in Figure 2.6

$$V_m = V_c \times 0.0283$$

$$V_a = V_m \frac{(P_a - P_m)}{P_a}$$

$$Q_1 = \frac{V_a}{t}$$

9. Record the barometric pressure in mm Hg.

10. After the 5-minute equilibration period, read the mercury manometer and record this value in column P_m. The example given in Figure 2.5 shows a reading of 70 millimeters (mm).

11. Read the water manometer and record this in column P_t. The example given in Figure 2.5 shows a reading of 3.0 inches.

12. Wind the stopwatch and set it in the horizontal position with the dial facing up.

13. Locate the uncompensated dial on the left end of the positive displacement meter. The location is shown in Figure 2.4. Note: this dial must be viewed from the end. One revolution of Dial #5 equals 10 cubic feet of air passed through the positive displacement meter.

14. Use a stopwatch to measure the time in minutes and hundredths of minutes for exactly 10 revolutions of Dial #5 (i.e., for 100 ft^3 of air to pass through the positive displacement meter). Record 100 under column V_c and the elapsed time under column t.

15. Record 2.83 under column V_m to convert cubic feet to cubic meters.

$$V_m = V_c \times 0.0283$$

$$100\ ft^3 \times 0.0283\ \frac{m^3}{ft^3} = 2.83\ m^3$$

16. Turn the motor OFF.

17. Repeat this procedure with each of the other load plates in the set.

18. Repeat Steps 1-17 one time.

19. Calculate and record V_a for each run.

$$V_a = V_m \frac{(P_a - P_m)}{P_a}$$

20. Calculate and record Q_1 for each run.

$$Q_1 = \frac{V_a}{t}$$

21. Plot a graph of Q_1 vs P_t on two-cycle, semi-log graph paper to obtain the hi vol orifice calibration line as illustrated in Figure 2.7. Use a straight edge to draw a best fit line through the calibration points.

22. If any calibration point does not fall within ± 1 percent of the line, rerun that point, recalculate, and replot. The percent deviation can be calculated by taking the questionable flowrate (Q_o) and the calibration line flowrate (Q_c) for the same P_t reading.

$$\text{Percent Deviation}^a = \frac{(Q_o - Q_c)}{Q_c} \times 100$$

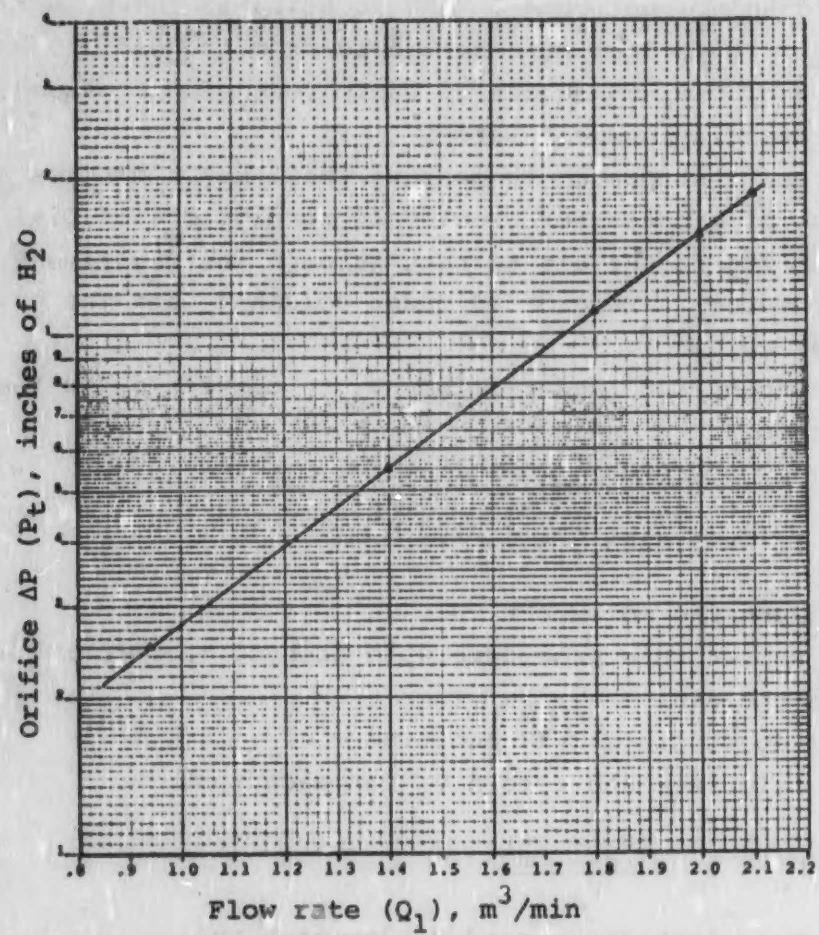


Figure 2.7 Example of hi vol orifice calibration relationship

---

[a] not to exceed ± 1 percent

2.6 Sampler

Samplers must be calibrated when first purchased, after major maintenance on the sampler (e.g., replacement of motor or motor brushes), any time the flow-rate measuring device (i.e., rotameter or recorder) has to be replaced or repaired, or any time a one-point audit check (see Section 2.2.8) deviates more than ±7% from the calibration curve.

In using the orifice calibration unit to calibrate a sampler, corrections must be made to the indicated flow rate if the ambient barometric pressure or temperature is substantially different from the pressure or temperature values recorded when the orifice unit was calibrated. Guidelines are given in Subsections 2.6.1 and 2.6.2 as to when the indicated flow rate should be corrected. Calculate the corrected flow rate as follows:

$$Q_2 = Q_1\left(\frac{T_2P_1}{T_1P_2}\right)^{1/2}$$

where

Q_2 = corrected flow rate at sampling conditions, m^3/min;

Q_1 = uncorrected flow rate read from the orifice unit calibration curve for a given pressure, in. H_2O;

T_1 = absolute temperature when orifice unit was calibrated, ^oK;

P_1 = barometric pressure when orifice unit was calibrated, mmHg;

T_2 = absolute temperature while calibrating the sampler, ^oK, and

P_2 = barometric pressure while calibrating sampler, mmHg.

For example, if $P_1 = P_2$, Figure 2.8 shows the percentage change of Q versus temperature differences. If T_2 is greater than T_1, the percentage change is positive; if T_2 is less than T_1, the percent change is negative. The same procedure is used to correct for pressure differences. The above formula corrects for field conditions only.

If data reporting requirements specify standard conditions, the following equation should be applied to the hi-vol flow rate to determine the total volume of air sampled.

$$V_s = Q_2 t\left(\frac{P_2 T_1}{P_1 T_2}\right)$$

where

V_s = sample volume at 760 mmHg and 298 $^{\circ}$K
P_2 = atmospheric pressure at sampling conditions, mm Hg
P_1 = standard pressure, 760 mmHg
T_2 = ambient temperature at sampling conditions, $^{\circ}$K
T_1 = ambient temperature at standard conditions, 298 $^{\circ}$K
t = Time, minutes

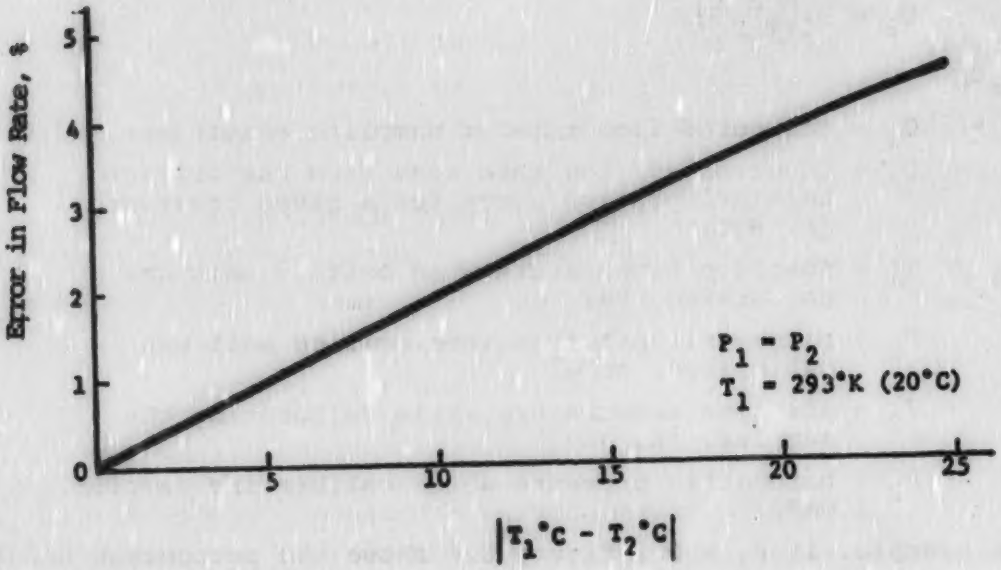


Figure 2.8 Percent change in flow rate versus temperature variations (orifice calibration unit)

2.6.1 Calibration of Hi Vol Sampler Without Flow Recorder - Figure 2.9 shows the apparatus required for the calibration of a high volume sampler in the field. The apparatus was arranged in this manner for illustration purposes only. Specifically, care should be taken not to restrict the air flow into the orifice unit or out of the motor unit.

All data during the initial and final calibration for one hi vol are recorded on the same page in the Hi Vol Calibration Log. See Figure 2.10 for an example and Section 2.2.12 (Data Forms) for a blank copy for use by the Handbook user. If the sampler is being calibrated for the first time or if it has been serviced, record the data on the "Initial" section of the calibration log. If the sampler is being calibrated prior to scheduled maintenance, record the data under the "Final" section of the calibration log.

The following stepwise calibration procedure is presented:

1. Assemble a hi vol with a clean filter and operate it for at least 5 minutes at normal line voltage. If a stepdown transformer is used during normal operation, then calibration should be performed with the transformer in operation.

2. Record the rotameter number and date on three gummed labels. Affix one gummed label to the very top of the metal part on the front of the rotameter. Affix another gummed label to the middle of the vacuum hose and affix the last gummed label to the side of the hi vol motor.

3. Attach the rotameter to the hi vol motor as shown in Figure 2.9.

4. Adjust the rotameter so the center of the ball reads 60. Figure 2.11 is a photo of the working components of the rotameter.

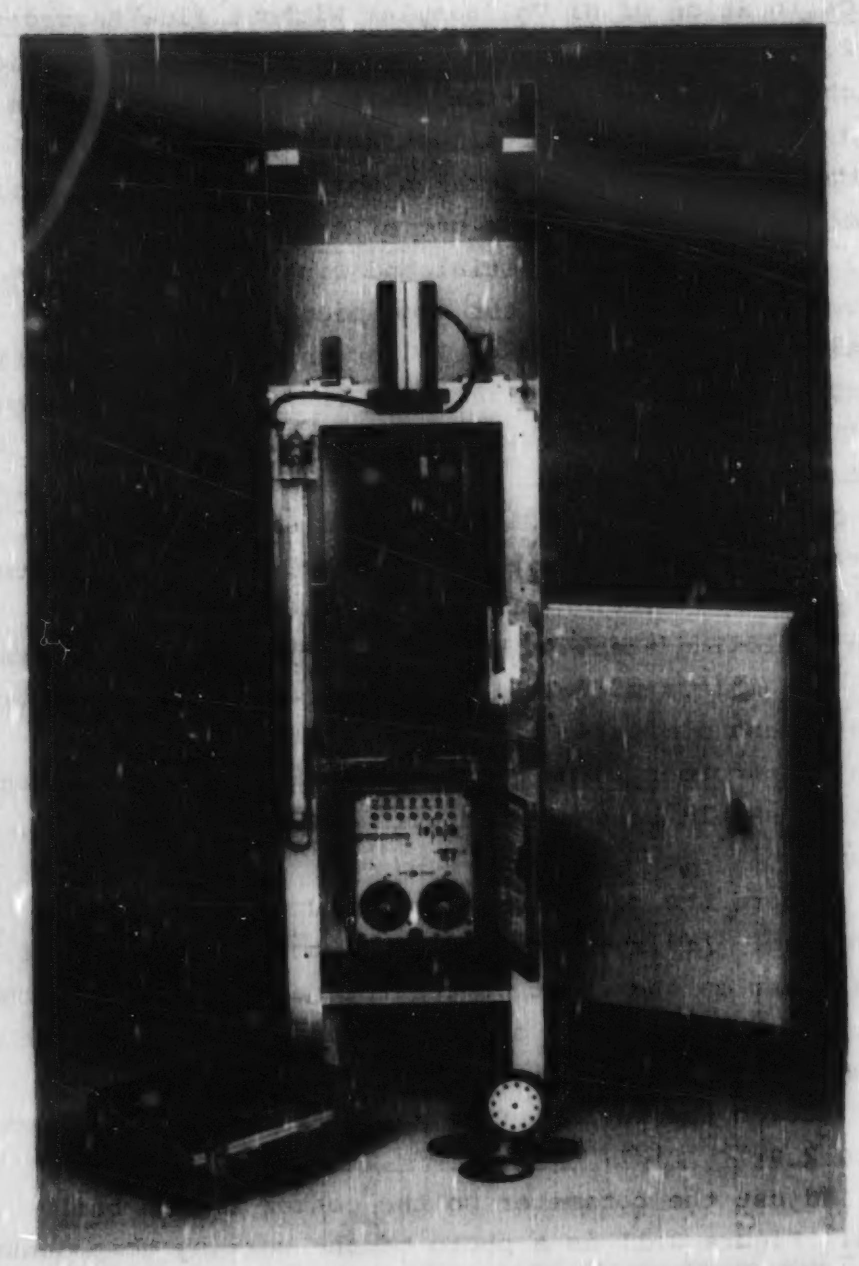


Figure 2-9. Hi vol and orifice assembled for calibration with rotameter.

Rotameter Number 16491
Motor Number 74891
Site Name Water Tower

	Initial				Final			
Description Derivation / Plate Number	Recorder or Rotameter Reading	Pressure drop Across the Orifice in inches of water	m^3/minute Flowrate Q_1	Corrected* Flowrate Q_2	Recorder or Rotameter Reading	Pressure drop Across the Orifice in inches of water	m^3/minute Flowrate Q_1	Corrected Flowrate Q_2
5	29.5	2.2	0.88	0.84	29.5	2.1	0.84	0.81
7	35.5	3.0	1.05	1.00	35.5	2.8	1.00	0.97
13	40.5	3.8	1.19	1.14	40.5	3.5	1.15	1.10
15	45.5	4.4	1.27	1.22	45.5	3.9	1.20	1.16
18	48.5	6.7	1.42	1.36	49.0	5.0	1.34	1.30

Date 7-1-75 Time 2:30pm. Temperature 20°C
Barometric Pressure 687 Orifice Number 4861 Voltage 115
Signed B Duncan Verified By Joe Smith

Date 9-15-75 Time 1:00 pm. Temperature 19°C
Barometric Pressure 701 Orifice No. 4861 Voltage 115
Signed B. Duncan Verified By Joe Smith

$$^*Q_2 = Q_1\left(\frac{T_2P_1}{T_1P_2}\right)^{1/2}$$

Figure 2.10 Example of Hi Vol Calibration Log

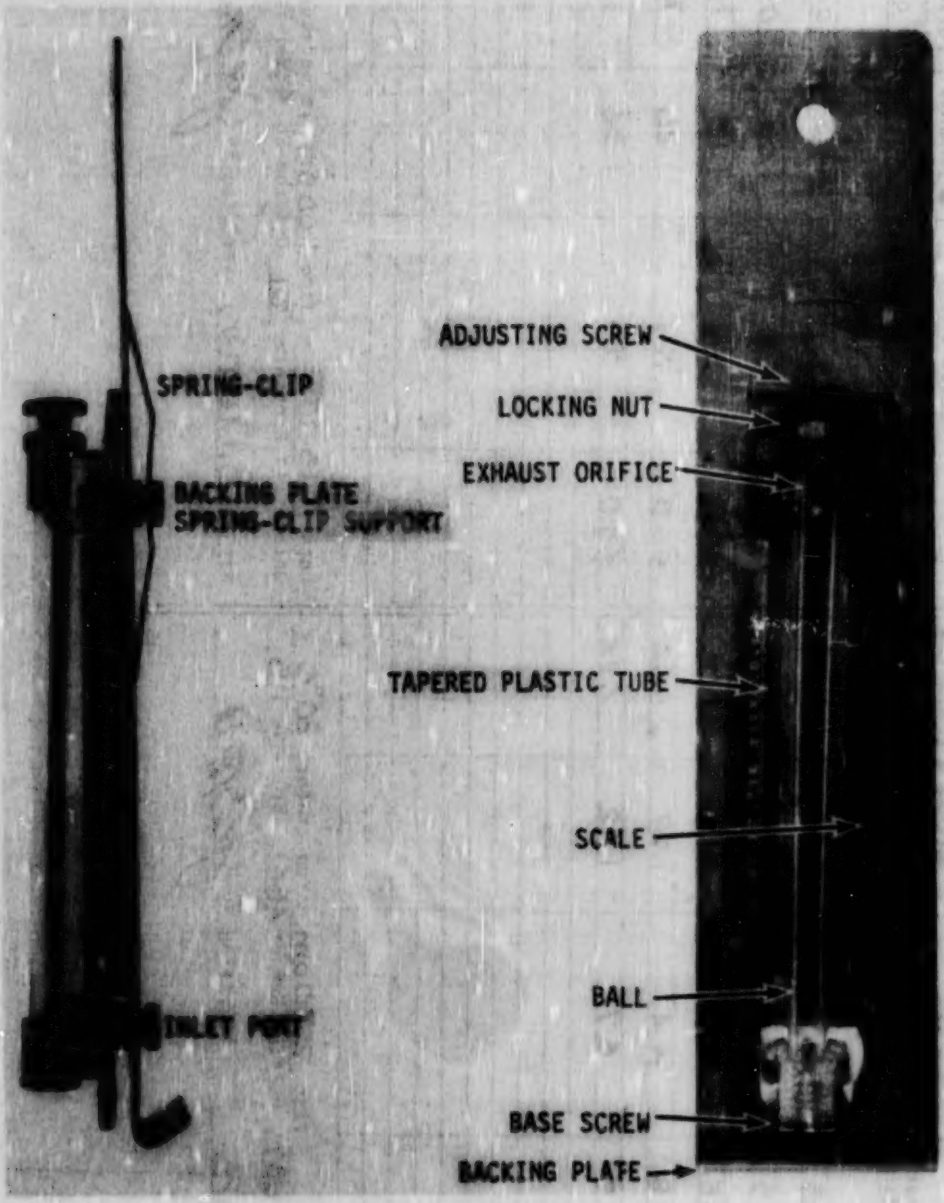

SPRING-CLIP
BACKING PLATE
SPRING-CLIP SUPPORT
INLET PORT
ADJUSTING SCREW
LOCKING NUT
EXHAUST ORIFICE
TAPERED PLASTIC TUBE
SCALE
BALL
BASE SCREW
BACKING PLATE

Figure 2-11. Example of hi vol rotameter.

- a. Hold the rotameter vertically.
- b. To adjust, loosen the locking nut by turning from left to right as seen from the front or counterclockwise as seen from above.
- c, Turn adjusting screw clockwise to lower the ball or counterclockwise to raise the ball.
- d. When the adjustment is made, tighten the locking nut making sure the ball continues to read 60.
- e. Seal both the locking nut and the adjustment screw with hot melt glue to assure the setting is not changed. Do not cover the exhaust orifice.
- f. Turn the hi vol motor OFF.

5. Remove the filter holder.

6. Attach the calibrated orifice with one of the load plates between the motor and the orifice.

7. Turn the motor ON and record the water manometer and rotameter readings after they stabilize.

8. Turn the motor OFF.

9. Repeat Steps 6-8 above for each of the other load plates.

10. Repeat Steps 6-9 once.

11. Determine and record the air flow rate as read from the hi vol orifice calibration curve for each manometer reading.

12. Record the barometric pressure in mm Hg and temperature in °C.

13. Determine the percent difference between the temperature (°C) and barometric pressure (mm Hg) recorded and the temperature and barometric pressure measured when the hi vol orifice was calibrated.

- a. If the recorded barometric pressure is within ±15 percent range and the recorded temperature is within ±100 percent range, proceed to step 16.
- b. If the barometric pressure exceeds the ±15 percent range or the temperature exceeds the ±100 percent range or both occur proceed to step 14.

14. Convert the temperature measured when the hi vol orifice was calibrated (T_1) and the temperature recorded in Step 12 above (T_2) to absolute temperature (°K).

15. Determine the true flow rates corrected to the barometric pressure and temperature recorded in Step 12 above. This is done by substituting each of the flow rates determined in Step 15 above for Q_1 in the following equation and solving for Q_2.

$$Q_2 = Q_1\left(\frac{T_2P_1}{T_1T_2}\right)^{1/2}$$

where

Q_1 = flow rate determined in Step 15 above
Q_2 = corrected flow rate
P_1 = barometric pressure measured when the hi vol orifice was calibrated
P_2 = barometric pressure recorded in Step 12 above
T_1 = absolute temperature determined at the time the hi vol orifice was calibrated, °K
T_2 = absolute temperature determined from Step 12 above, °K.

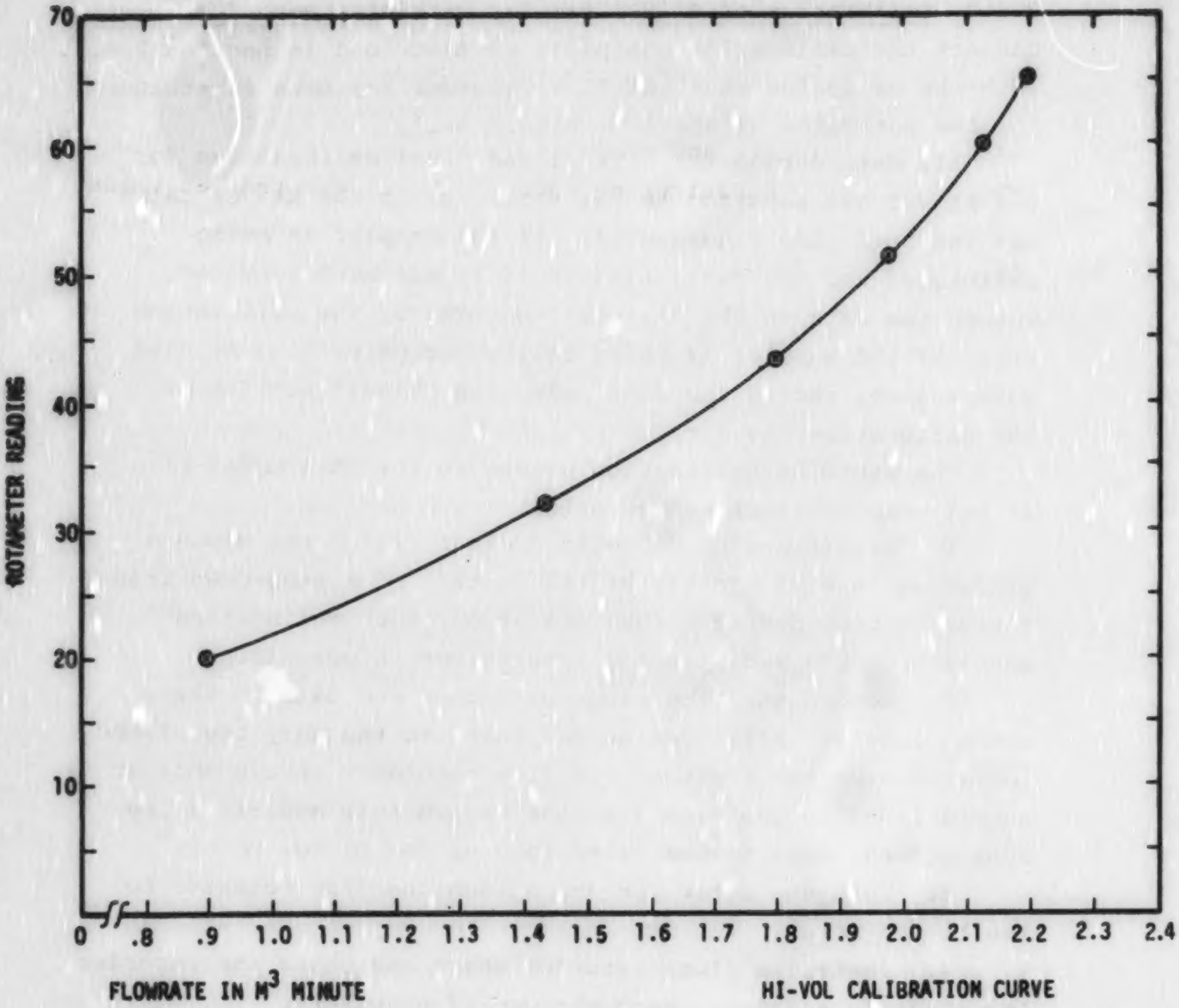
16. Plot the rotameter readings vs. the air flow rates as shown in Figure 2.12.

17. Use a French curve to draw a best fit smooth curve through the calibration points.

18. If any calibration point does not fall within ± 5 percent of the curve, or causes the curve to be S-shaped or have a sharp turn, rerun that point, recalculate, and replot. The percent deviation can be calculated by taking the questionable flow rate (Q_o) and the calibration curve flow rate (Q_c) for the same rotameter reading.

$$\text{Percent deviation} = \frac{(Q_o - Q_c)}{Q_c} \times 100$$

should fall within ± 5 percent.



Temperature 25°
Barometric Pressure 760mm
Calibration log page no. 18
Hi Vol. No. 5
Date February 16, 1976
Analyst B.J. Smith

Figure 2.12 Example of hi vol calibration curve

2.6.2 Calibration of Hi Vol Sampler with Flow Recorder - Connect the calibration equipment as described in Section 2.6.1, with the exception that the flow recorder has been substituted for the rotameter as shown in Figure 2-13.

All data during the initial and final calibrations for one hi vol are recorded on the same page in the Hi Vol Calibration Log. See Figure 2.10. If the sampler is being calibrated for the first time or if it has been serviced, record the data on the "Initial" section of the calibration log. If the sampler is being calibrated prior to scheduled maintenance, record the data under the "Final" section of the calibration log.

The stepwise calibration procedure for this model of Hi Vol sampler is presented below:

1. Assemble a hi vol with a clean filter and operate it for at least 5 minutes at 115 volts. If a step-down transformer is used during normal operation, then calibration should be performed with the transformer in operation.

2. Record the flow recorder number and date on three gummed labels. Affix one gummed label to the very top of the metal face on the front of the flow recorder. Affix another gummed label to the middle of the vacuum hose and affix the last gummed label to the other side of the hi vol motor.

3. Turn the motor OFF and attach the flow recorder to the hi vol motor.

4. Install a clean recorder chart and check the recorder for proper operation. Zero the pen if necessary.

5. Remove the filter holder.

6. Attach the calibrated orifice with one of the load plates between the motor and the orifice.

7. Turn the motor ON and record the water manometer and flow recorder readings after they stabilize.

8. Turn the motor OFF.

9. Repeat Steps 6-8 for each of the other load plates.

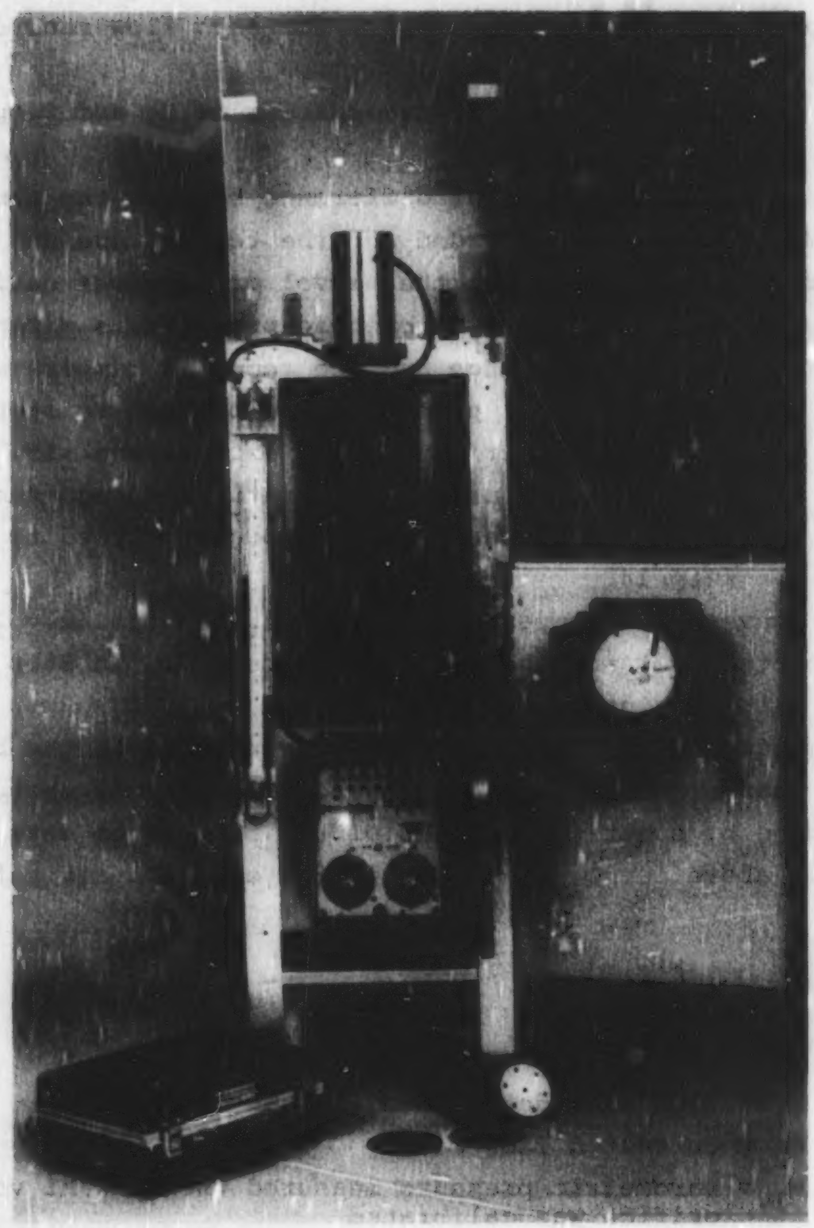


Figure 2-13. Hi vol and orifice assembled for calibration with flow recorder.

10. Repeat Steps 6-9 once.

11. Determine and record the air flow rate as read from the hi vol orifice calibration curve for each flow recorder reading.

12. Record the barometric pressure in mm Hg and temperature in °C.

13. Determine the percent difference between the temperature and barometric pressure recorded and the temperature and barometric pressure measured when the hi vol orifice was calibrated.

  a. If the recorded barometric pressure is within ± 15 percent range and the recorded temperature is within ± 100 percent range proceed to step 16.

  b. If the barometric pressure exceeds the ± 15 percent range or the temperature exceeds the ± 100 percent range or both occur proceed to step 14.

14. Convert the temperature measured when the hi vol orifice was calibrated (T_1) and the temperature recorded in Step 12 above (T_2) to absolute temperature (°K).

15. Determine the true flow rates corrected to the barometric pressure and temperature recorded in Step 12 above. This is done by substituting each of the flow rates determined in Step 15 above for Q_1 in the following equation and solving for Q_2.

$$Q_2 = Q_1\left(\frac{T_2P_1}{T_1P_2}\right)^{1/2}$$

where

Q_1 = flow rate determined in Step 15 above
Q_2 = corrected flow rate
P_1 = barometric pressure measured when the hi vol orifice was calibrated
P_2 = barometric pressure recorded in Step 12 above
T_1 = absolute temperature determined at the time the hi vol orifice was calibrated, °K
T_2 = absolute temperature determined from Step 12, °K.

16. Plot the flow recorder readings vs. the air flow rates.

17. Use a French curve or a curve fitting technique such as the least squares fit, to draw a best fit smooth curve through the calibration points.

18. If any calibration point does not fall within ± 5 percent of the curve, or causes the curve to be S-shaped or have a sharp turn, rerun that point, recalculate, and replot. The percent deviation can be calculated by taking the questionable flow rate (Q_o) and the calibration curve flow rate (Q_c) for the same flow meter reading.

$$\text{Percent deviation} = \frac{(Q_o - Q_c)}{Q_c} \times 100$$

should fall within ± 5 percent.

Table 2.1 ACTIVITY MATRIX FOR CALIBRATION OF EQUIPMENT

Equipment	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Analytical balance	3 to 5 standard weights covering normal range of filter weight, all with indicated weight = true weight ± 0.0005 g	Gravimetric test weighing at purchase and when performing periodic calibration checks	Have balance maintained and calibrated by manufacturer's representative
Relative humidity indicator	Indicator reading = psychrometer reading ± 6%	Comparison with reading of wet-bulb dry-bulb psychrometer on receipt and at 6-month intervals	Adjust or replace to attain acceptance limits
Timer	±15 minutes/24 hours	Check at purchase and quarterly against elapsed time meter	Adjust and repeat test
Elapsed time meter	±2 minutes/24 hours	Standard timepiece (known accuracy) at receipt and at 6-month intervals	Adjust or replace time indicator to attain acceptance limits
Orifice calibration unit	Indicated flow rate = actual flow rate ± 4%	Flow rate primary standard at receipt and 1-year interval	1) Adopt new calibration curve if evidence of orifice damage is absent 2) Replace orifice unit if evidence of damage is present
Sampler	$\frac{\lvert Q_o - Q_c \rvert}{Q_c} \le 0.05$ Q_o = observed flow rate Q_c = flow rate from calibration curve	Calibration orifice unit, on receipt and after major maintenance on sampler	Rerun points for which $\frac{\lvert Q_o - Q_c \rvert}{Q_c} > 0.05$ until acceptance limit attained

3.0 FILTER SELECTION AND PREPARATION

Suppliers of glass fiber filters for the measurement of TSP have available two grades of filter materials, the normal or traditional filter grade, which has been in use for over 20 years, and a spectro-quality grade. The spectro-quality grade contains less organic and inorganic contaminants. Spectro-quality grade filters are recommended for use in those applications where additional chemical analyses are anticipated. A filter with low surface alkalinity is recommended to avoid positive interferences from absorption of acid gases while sampling. Ideally, surface alkalinity should be between pH 6.5 and 7.5. However, most glass fiber filters commercially available at the present time have a pH greater than 7.5. An activity matrix for filter selection and preparation is presented in Table 3.1 at the end of the section.

3.1 Filter Selection

Only filters having a collection efficiency of at least 99 percent for particles of 0.3-μm diameter, as measured by the DOP test (ASTM-D2986-71), are to be used. The manufacturer should be required to furnish proof of the collection efficiency of a batch of new filters when purchased. Record in procurement log, Figure 1.1.

Each filter must be visually inspected with the aid of a light table. Discard or return to the supplier filters with pinholes and other defects such as tears, creases or lumps. Remove loose particles with a soft brush.

3.2 Filter Identification

Assign a serial number to each filter. Stamp this number on two diagonally opposite corners, one stamp on each side of the filter. Apply gentle pressure to avoid damaging the filter.

3.3 Filter Equilibrium

Equilibriate the filter in the conditioning environment for 24 hours prior to weighing to minimize errors in measuring the weight; longer periods of equilibrium will not affect accuracy. The conditioning environment should have an average temperature between 20° and 25°C and vary not more than ±3°C, and a relative humidity (RH) below 50 percent and vary not more than ±5 percent. A convenient working RH is 40 percent.

3.4 Filter Weighing

Clean filters are usually processed in lots, that is, several at one time. Before weighing the first filter, perform a balance check by weighing a standard Class "S" weight of between 3 and 5 grams. Record the actual and measured weights, the date, and the operator's initials, as shown in Figure 2.1. If the actual and measured values differ by more than ±0.5 mg (0.0005 g), report it to the supervisor before proceeding.

If the actual and measured values agree to within ±0.5 mg, weigh each filter to the nearest mg. Weigh each filter within 30 seconds after removal from the equilibration chamber, record the tare weight and serial number of each filter in the Laboratory Data Log, Figure 3.1 Section 2.2.12 (Data Forms) contains a blank copy of Figure 3.1 for use by the Handbook user. Clean filters must not be folded or creased prior to weighing or use.

3.5 Filter Handling

It is recommended that a quantity of filters sufficient for at least a three (3) month period for each sampler be numbered and weighed at one time. Pack the filters in their original container or a box of similar size such that each filter is separated by a sheet of 8 1/2- by 11-inch tracing paper. Be sure that the filters are stacked in the box in numerical order so that the operator will use the proper filter first. In addition to the filters, supply the field operator with pre-addressed return envelopes to protect the filters during mailing. These folders can also be printed front and back to serve as a sample record data sheet as shown in Figure 3.2 Section 2.2.12 (Data Forms) contain a blank copy of Figure 3.2 for use by the Hanbdook user.

TOTAL SUSPENDED PARTICULATE

NAME PARAMETER CODE 1 1 1 0 1

Notown, Ohio
CITY NAME

600 South Street
SITE ADDRESS

PROJECT

TIME INTERVAL

STATE ☐☐ AREA ☐☐☐☐ SITE 371

PROJECT ☐☐ TIME ☐ YEAR 75

INSTRUMENT LAST CALIBRATED 6/4/75

SAMPLER IDENTIFICATION NO. 03261

SAMPLING DATE MONTH	SAMPLING DATE DAY	SAMPLING DATE ST HR	CONC µg/m³	FILTER NUMBER	AVERAGE * CFM	SAMPLING TIME	AIR m³ VOLUME	GROSS WGT.	TARE WGT.	NET WGT.	REMARKS
07	08	00	797	10004	49.4	1437	19934	3.301	3.142	0.159	

*FROM AVERAGE FLOW DETERMINED FROM CALIBRATION

LABORATORY FUNCTIONS

FILTER PREPARATION

- FILTER MUST BE FREE OF PIN-HOLES, TEARS, CREASES, LUMPS, ETC.
- FILTER MUST BE PROPERLY EQUILIBRATED (20 hrs< TIME<28 hrs; HUMIDITY<50%)
  (Δ HUMIDITY ± 5%; Δ TEMP ± 3°C)
- BALANCE MUST BE CHECKED BEFORE WEIGHING (STD. WGT. 3 to 5 g ± 0.0005 g)

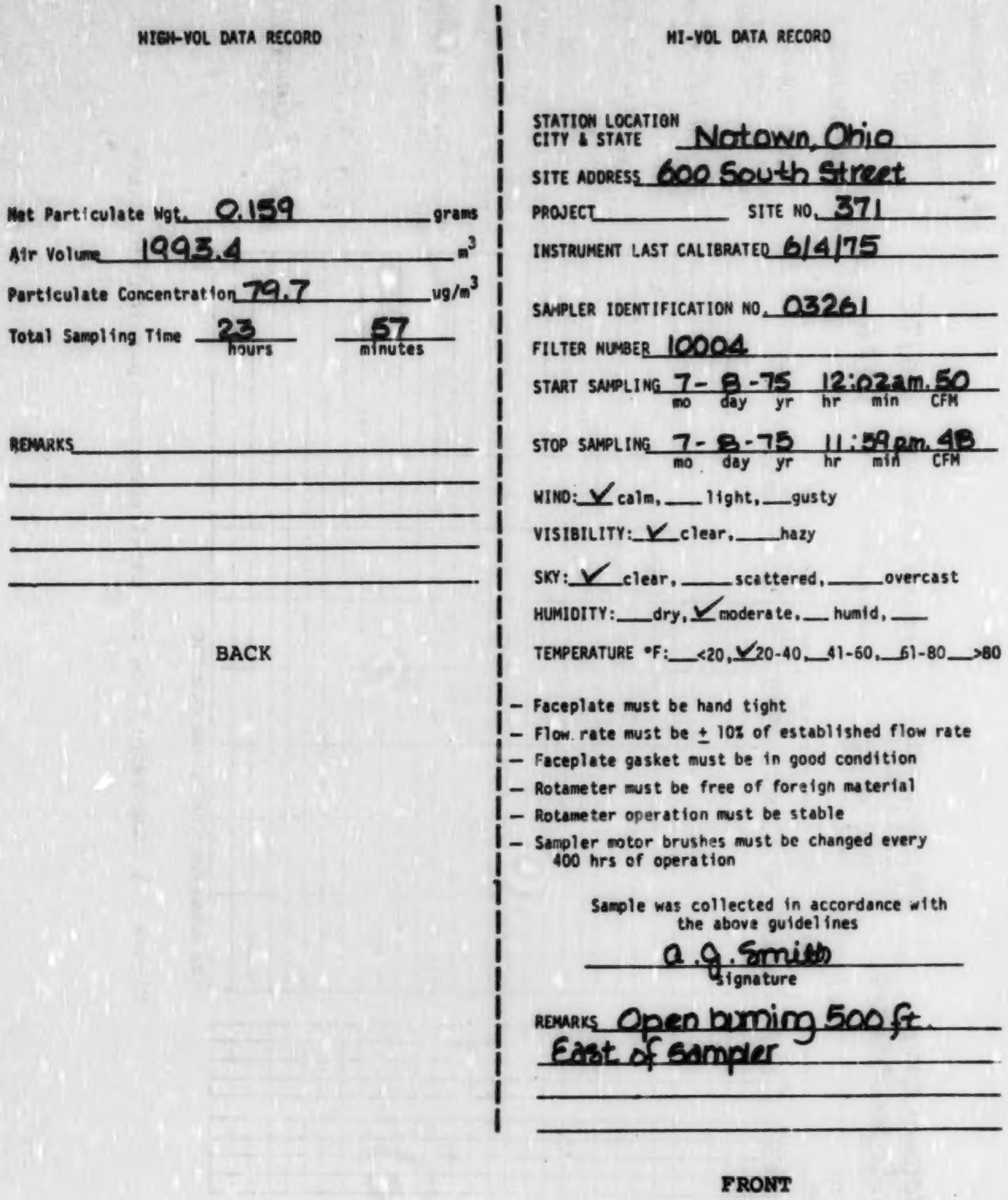
SAMPLING ANALYSIS

- SAMPLE MUST BE FREE OF EVIDENCE OF MALFUNCTION; TEN OR FEWER INSECTS IN SAMPLE
- FILTER MUST BE PROPERLY EQUILIBRATED (SEE ABOVE)
- BALANCE MUST BE CHECKED BEFORE WEIGHING (SEE ABOVE)

ALL SAMPLES WERE HANDLED IN ACCORDANCE WITH THE ABOVE GUIDELINES

A. G. Smith
SIGNATURE

Figure 3.1 Laboratory log for total suspended particulate data



HIGH-VOL DATA RECORD

Net Particulate Wgt. 0.159 grams

Air Volume 1993.4 m^3

Particulate Concentration 79.7 ug/m^3

Total Sampling Time 23 hours 57 minutes

REMARKS

BACK

HI-VOL DATA RECORD

STATION LOCATION CITY & STATE Notown, Ohio

SITE ADDRESS 600 South Street

PROJECT ___ SITE NO. 371

INSTRUMENT LAST CALIBRATED 6/4/75

SAMPLER IDENTIFICATION NO. 03261

FILTER NUMBER 10004

START SAMPLING 7- 8 -75 12:02am. 50 (mo day yr hr min CFM)

STOP SAMPLING 7- 8 -75 11:59pm. 48 (mo day yr hr min CFM)

WIND: [✓ calm], ___ light, ___ gusty

VISIBILITY: [✓ clear], ___ hazy

SKY: [✓ clear], ___ scattered, ___ overcast

HUMIDITY: ___ dry, [✓ moderate], ___ humid, ___

TEMPERATURE °F: ___ <20, [✓ 20-40], ___ 41-60, ___ 61-80 ___ >80

- Faceplate must be hand tight
- Flow rate must be ± 10% of established flow rate
- Faceplate gasket must be in good condition
- Rotameter must be free of foreign material
- Rotameter operation must be stable
- Sampler motor brushes must be changed every 400 hrs of operation

Sample was collected in accordance with the above guidelines

a. g. Smith
Signature

REMARKS Open burning 500 ft. East of sampler

FRONT

Figure 3.2 Hi vol field data sheet

Table 3.1 ACTIVITY MATRIX FOR FILTER SELECTION AND PREPARATION

Activity	Acceptance limits	Frequency or method of measurement	Action if requirements not met
Filter selection and collection efficiency	1) 0.3 µm diameter particle collection efficiency > 99% 2) No pinholes, tears, creases, etc.	1) Manufacturing should furnish proof of DOP test (ASTM-D2986-71) 2) Visually check each filter with aid of light table	1) Reject shipment 2) Return to supplier
Filter identification	Identification number in accordance with specifications	Visually check each filter	Identify properly or discard filter
Filter equilibration	Equilibration in controlled environment for no less than 24 hours. Room: Constant humidity chamber employing a saturated chemical solution to afford an RH of less than 50% and constant within ±5%. Average temperature between 20 and 25°C with variation less than ±3%.	For each sample observe: • room or chamber conditions • equilibration period	Repeat equilibration step after 48 hours or more at ambient conditions
Filter weighing	Determine indicated filter weight to nearest milligram within 30 sec. after removal from equilibration environment	Gravimetric	Reweigh after 48 hours or more at ambient conditions and repeat of equilibration step
Filter handling	Filter in protective folder and envelopes undamaged	Visually check each filter	Repack undamaged filters, discard damaged filters

4.0 SAMPLING PROCEDURE

An activity matrix for sample collection functions is presented in Table 4.1 at the end of this section. The matrix summarizes the sample collection activities and the operational checks described in this section.

4.1 Installation of Filter

Place the sampler in the servicing position, Figure 4.1, by raising the sampler until the filter holder is above the top level of the shelter; then rotate the unit one-eighth turn so that it hangs from the top of the filter holder. Figure 4.2 shows the normal operating configuration of the unit. During inclement weather, including high winds, the entire sampler should be removed to a protected location for servicing whenever practicable. The clean, installed filter should be protected when returning the sampler to the shelter. The use of a filter cartridge assembly (available from General Metal Works), see Figure 4.3, permits the filter installation to be performed without requiring the entire sampler to be moved.

While the sampler is removed from the shelter or before the new filter is installed, the inside surfaces of the shelter cover and the surfaces around the filter holder should be cleaned of loose particles by wiping with a clean cloth.

To install a filter, remove the faceplate of the sampler by loosening the four wing nuts and swinging the bolts outward, see Figure 4.4. Place the filter, rough side up, on the wire screen. Center the filter on the screen so that when the faceplate is in position, the gasket will form an airtight seal on the outer edge (1/2 inch) of the filter. When aligned correctly, the edges of the filter should be parallel both to the edges of the screen behind it and to the faceplate gasket above it. The results of poorly aligned filters appear as uneven white borders, Figure 4.5, around the filter.

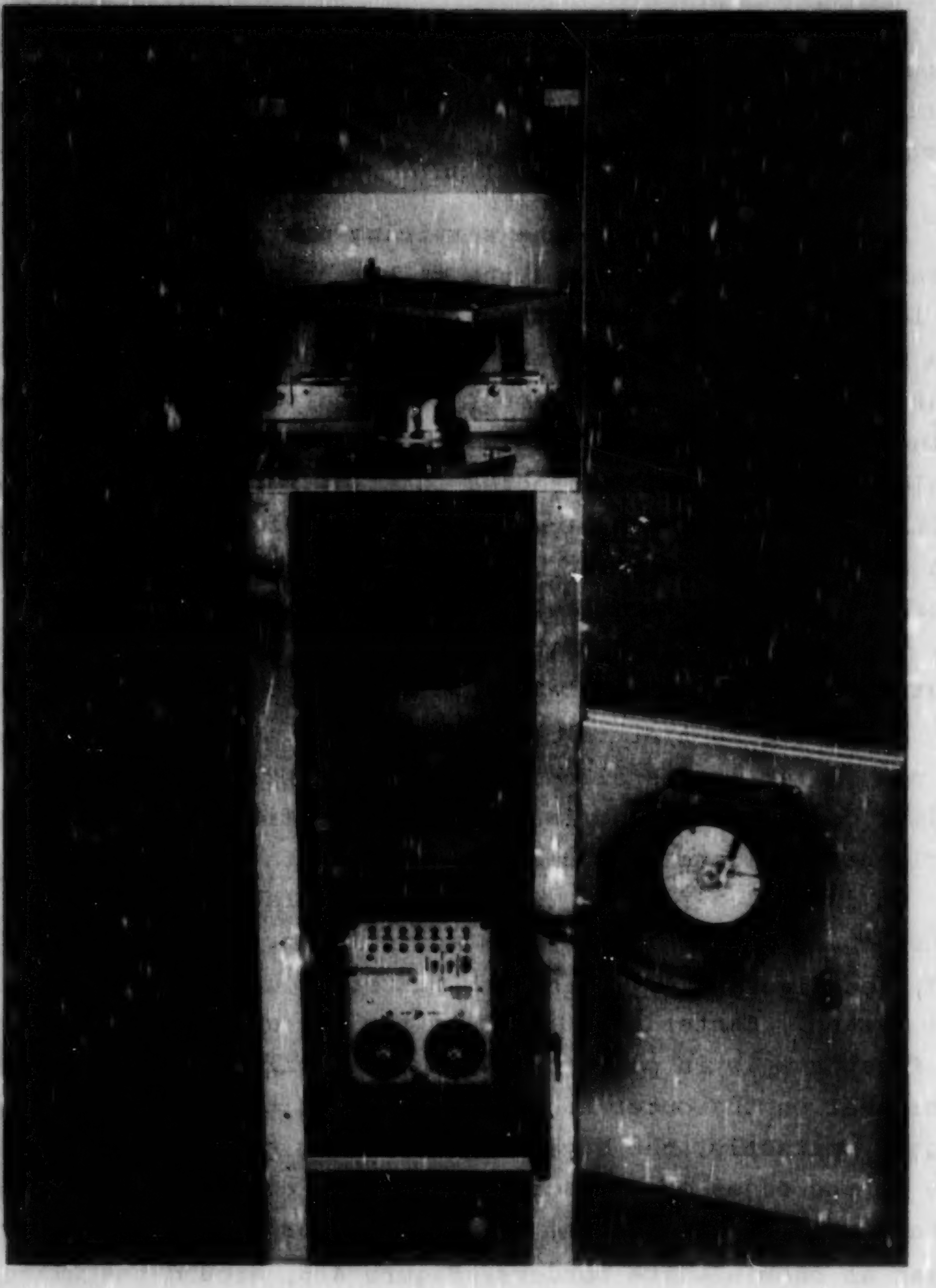


Figure 4-1. Hi vol in servicing position.

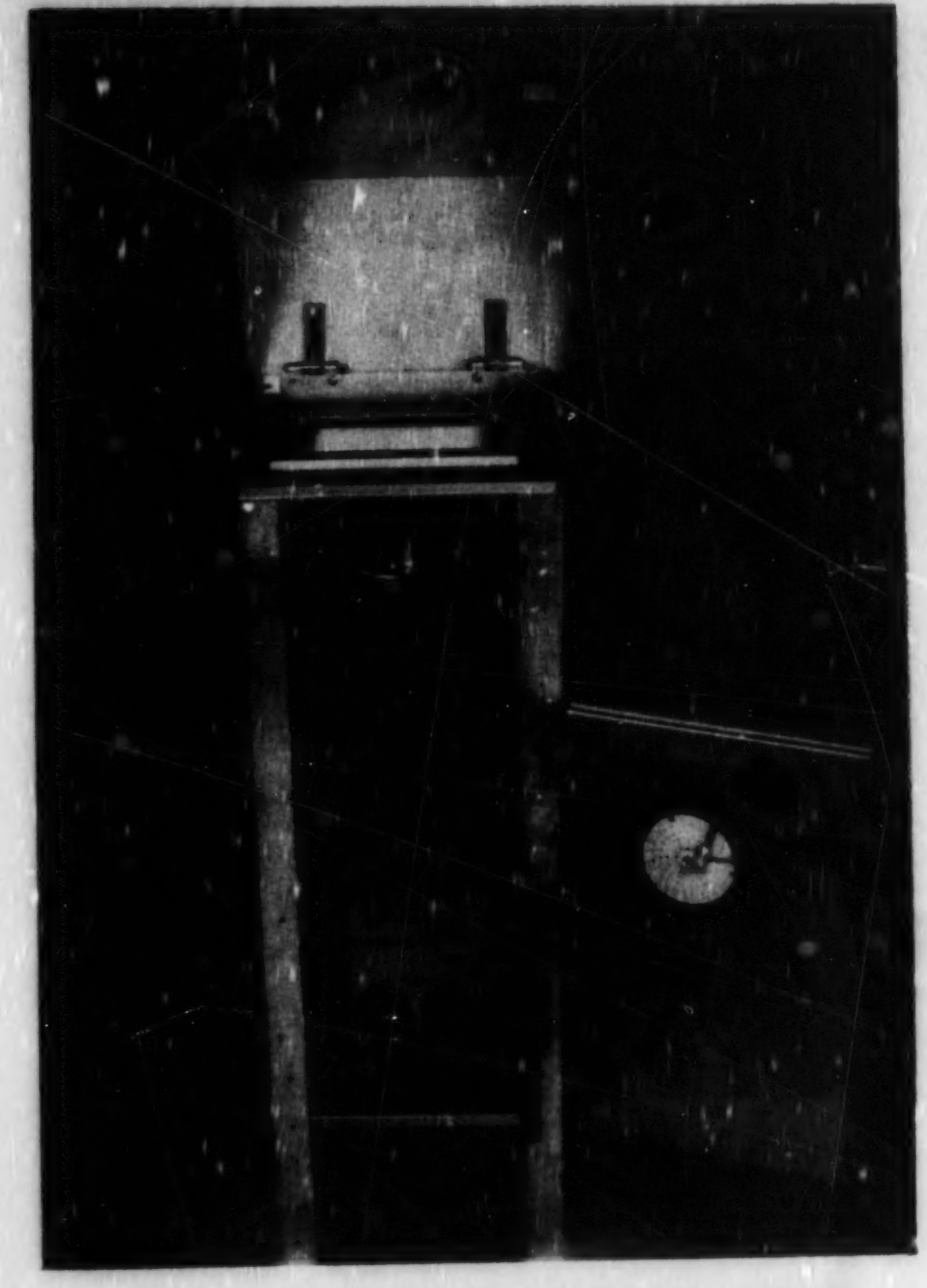


Figure 4-2. Hi vol in operational position.

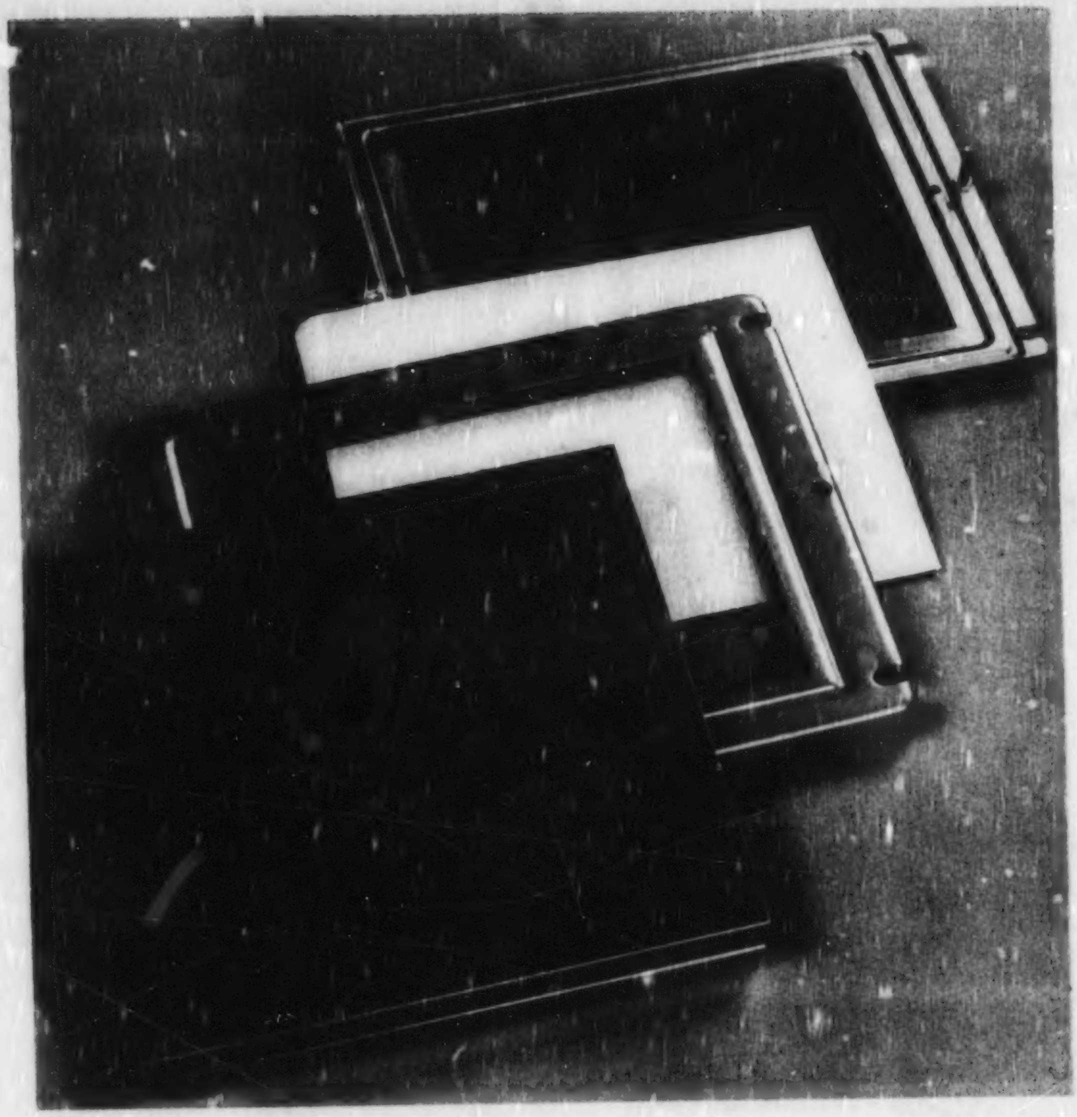


Figure 4-3. Hi vol filter cartridge assembly.

PART NO.	DESCRIPTION
F.H. 2100	FILTER HOOD COMPLETE
F.H. 2017	ALUMINUM HOLD DOWN FRAME
F.H. 2018	RUBBER GASKET 8" x 10"
F.H. 2021	WING NUT & BOLT WITH RIVET

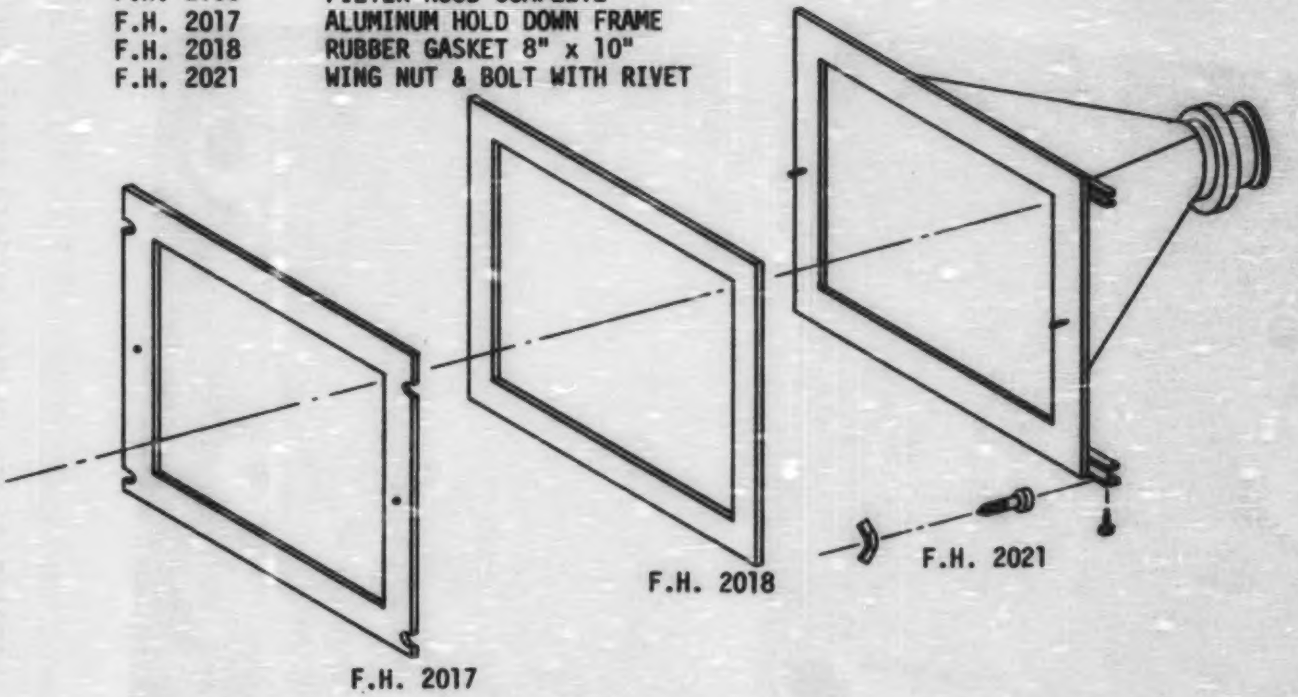


Figure 4.4. Diagram of hi vol sampler filter holder.

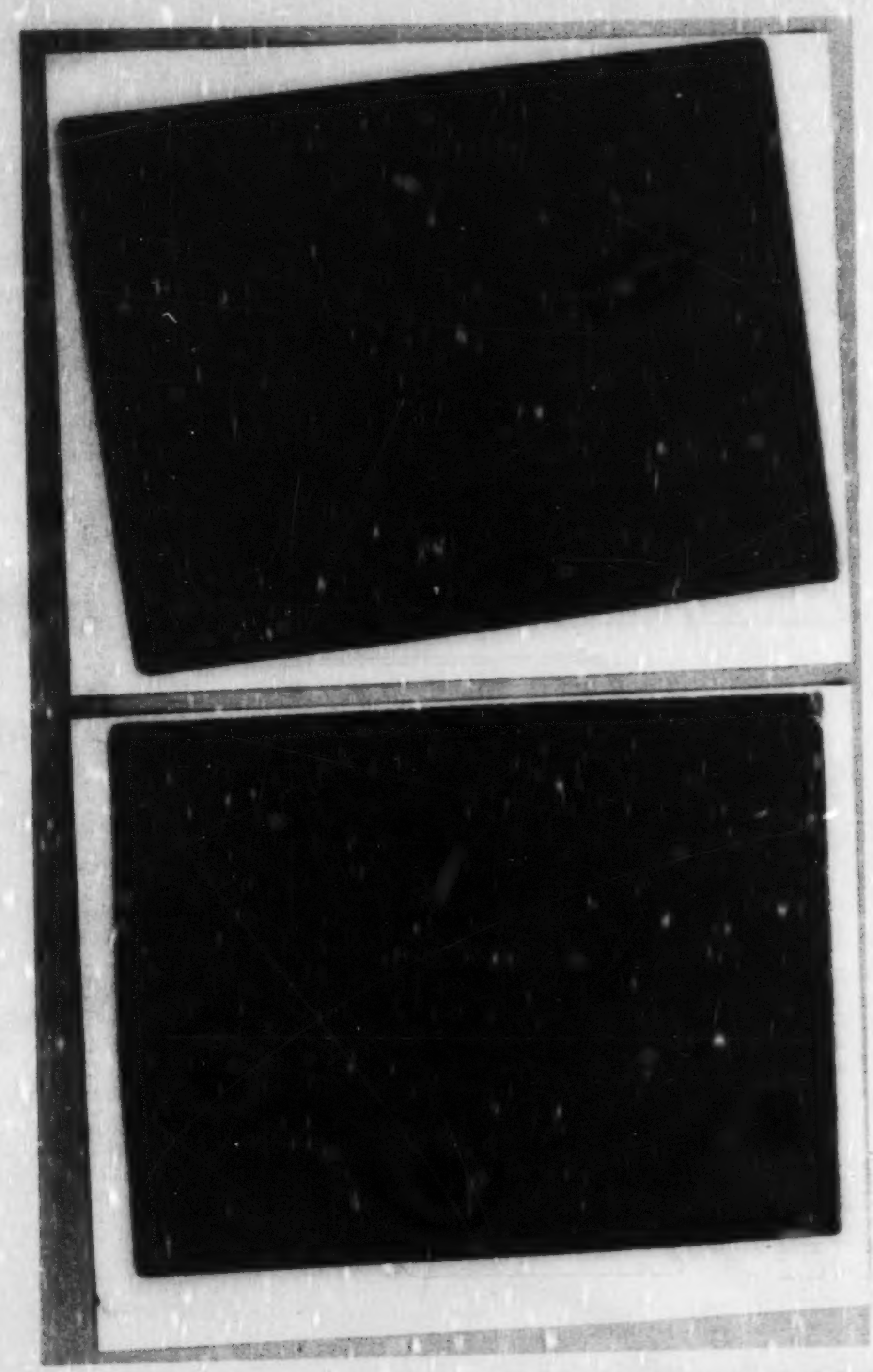


Figure 4-5. Examples of nonuniform borders resulting from poorly aligned filters.

When the filter is aligned and the faceplate is in place, the four wing nuts are tightened just enough to prevent leakage. Excessive tightening may cause the filter to stick to the gasket and result in permanent damage to the gasket.

The entire motor assembly (sampler) is rotated and lowered to its normal operating position, Figure 4.2.

4.2 Operational Checks

4.2.1 Flow-Rate Measurements with Rotameter Equipped Sampler -

Make flow-rate measurements while the sampler is at normal operating temperature. This requires a warmup time of at least 5 minutes before a valid measurement can be obtained.

Connect the rotameter to the sampler, using the same tubing as was used to calibrate, and place or hold in a vertical position at eye level. Read the widest part of the float. Use the calibration chart to convert the reading to cubic meters per minute rounded to the nearest 0.03 m^3/min., see Figure 2.12.

Flow rates are measured at the beginning and end of each sampling period. Additional measurements at different times during the sampling period may be required in special instances, such as in those cases where nonlinear loading on the filter occurs. For example if the sampler is located near a source which only operates for a specific period over the total 24-hour sampling period, flow measurements should be taken at the end of the specific period.

Precautions to be taken when making flow-rate measurements include:

1. After connecting the rotameter to the sampler, observe for at least one minute before taking a reading. If a gradual change in flow rate is observed do not take a reading until equilibrium is reached. A gradual change will usually be observed when the rotameter is at a substantially different temperature from the sampler exhaust air and may require 2 to 3 minutes to equilibrate.

2. Filter resistance and associated flow rate readings may change with time because of weather changes or changes in the condition of the sample deposited on the filter. If a clock switch is used to start and stop the sampler at preset times, flow rate measurements should be made within 30 minutes of the actual start and stop times to minimize discrepancies in in flow rate readings. If it is not practical to make the final flow reading within 30 minutes of stop time, the sampler should be turned ON and warmed up for 2 to 3 minutes prior to recording the final flow measurement.

4.2.2 Flow Rate Measurements on Flow Recorder Equipped Sampler - The following operational procedure is provided for use with Hi Vol sampler equipped with flow recorder.

1. Record on the backside of the new chart the filter number, station and sampler numbers, start time and date of start time.

2. Remove any moisture from inside the recorder case by wiping with clean cloth. Carefully insert the new chart into the recorder, being careful not to bend the pen arm beyond its limits of travel. An easy way to do this is to push in on the extreme top of the pen arm with the right hand to raise the pen head while inserting the chart with the left hand. A properly installed chart is shown in Figure 4.6. Be careful not to damage or weaken the center tab on the chart and make certain that the tab is centered on the slotted drive so that the chart will rotate the full 360 degrees in 24 hours with no binding or slippage.

3. Check to see that the pen head rests on zero (i.e., the smallest diameter circle on the chart). If it does not, tap the recorder lightly to make certain the pen arm is free; if it still does not read zero, adjust to zero with the adjustment screw (follow manufacturer's direction for specific recorder).

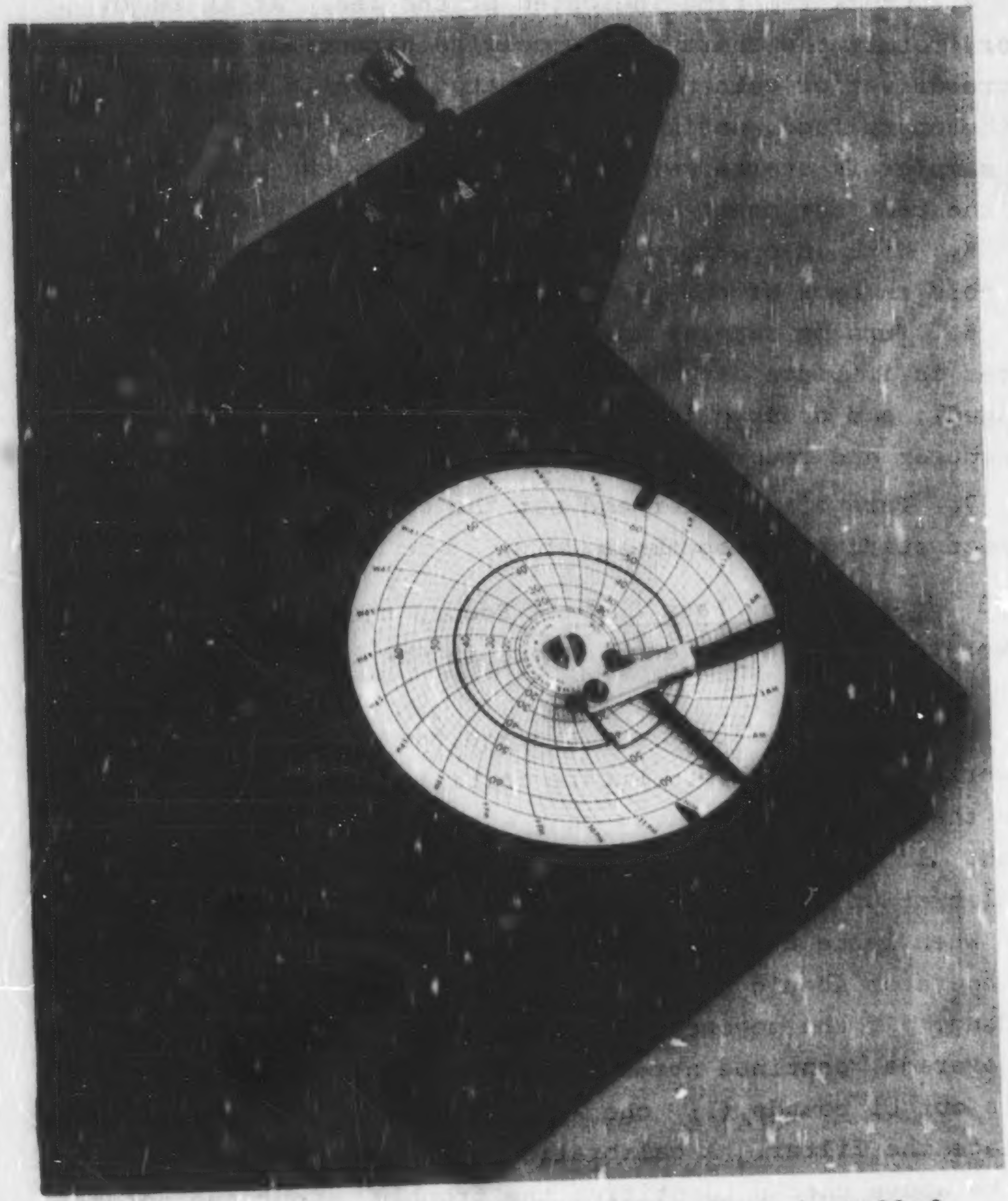


Figure 4-6. Flow rate recorder with chart installed.

4. Check the time indicated by the pen. If it is in error, rotate the chart in a clockwise direction, by inserting a screwdriver or coin into the slotted drive in the center of the chart face, until the correct time is indicated. If the sampler is started with a clock switch, the correct time for the recorder chart is the starting time on the clock switch.

5. With an eyedropper put a small amount of ink into the hole in back of the pen tip.

6. Turn on sampler (never turn on the sampler unless a filter is in place because the transducer and recorder may be damaged), and observe long enough to determine whether the transducer and recorder are operating properly.

7. Turn off sampler and set the clock switch for correct start and stop times.

4.2.3 Routine Flow Rate Check - Record the initial and final flow-rate readings for each sample in the log book maintained with the sampler.

After each calibration, average the first four initial flow-rate measurements. Future initial flow rates deviating more than ± 10 percent from this average should be investigated. If the change has been gradual over a period of time, a calibration is required.

When large deviations occur between successive samples, the operator should wait 5 minutes and make an additional reading. If the second reading falls within ± 10 percent of the average, continue normal operation. If the second reading falls out of bounds, (1) check the line voltage and/or (2) replace the filter. A calibration check is made if neither of the above checks identifies the trouble. Continue normal operations if the calibration check is satisfactory or perform a complete calibration if the check is unsatisfactory. Samplers equipped with a continuous recorder should be observed for at least 5 minutes before recording the initial flow rate.

The same procedure is used for final flow-rate measurements except that ± 20 percent of the average is initially used. Other valid limits may be determined for any sampling site as data are accumulated. A final flow rate deviating from the average by more than 20 percent may result from short-term weather changes. The occurrence of such conditions is noted on the data sheet for that sample. If a final flow rate less than 1.14 m^3/min (40 ft^3/min) is observed, the sample is voided, because at this low air flow the motor heats up and a valid flow-rate measurement cannot be obtained.

4.3 Time Measurements

Sampling period start and stop times, for samplers not equipped with a clock switch or an elapsed time meter, are recorded by the operators who start and stop the sampler, respectively. If different operators are involved, they set their watches to a common reference in order to arrive at an accurate sampling period time. Such a reference could be an office clock that is checked daily or the local telephone company giving time-of-day service.

Start and stop times for samplers equipped with a timer switch are taken from the timer's clock start and stop settings. The timer clock is checked, and set if necessary, for the correct time at each filter change. Because timer clocks cannot be set or read to less than ±15 minutes an elapsed time meter must be used to determine the number of minutes sampled. Elapsed time meters must be checked as described in Section 2.3 prior to being used.

Record the number of minutes of sample collection, determined by the operators watch or an elapsed time meter, on the hi vol field data sheet. The time of day which sample collection started or stopped is also to be recorded on Figure 3.2.

For samplers equipped with neither an elapsed time indicator nor a continuous flow rate recorder, the local power company should be contacted to see if the power has been off anytime during the sampling period. If so, record the length of time that the power was off and the source of the information on Figure 3.2, the remarks line.

Void samples when the sampling period is less than 23 hours or greater than 25 hours.

4.4 Sample Handling

4.4.1 Removing Exposed Filter - Place the sampler in the servicing position, Figure 4.1. Remove the faceplate and lift the exposed filter from the supporting screen by grasping it gently at the ends, not at the corners. With the exposed side in, fold the filter lengthwise at the middle, and if the collected sample is not centered on the filter (i.e., the unexposed border is not uniform around the filter) fold so that deposit touches only deposit. Results of an improperly folded filter are illustrated in Figure 4.7 where smudge marks from the deposit can be seen extending across the borders. This reduces the value of the sample for some analyses in which the collected sample must be subdivided into equal portions.

Place the filter in its numbered folder.

For samplers equipped with a flow rate recorder, remove the sample's recorder chart, record the stop time on the back side, and place the chart inside the filter folder with the inked side against the folder and the back side against the filter.

Place the folder containing the exposed filter and the flow recorder chart, if used, in a glassine envelope. This envelope is then placed in a mailing container if it is to be mailed.

Figure 4-7. Example of smudged filter border resulting from an improperly folded filter.

4.4.2 Routine Checks - The following listed checks should be made when removing an exposed filter:

1. Check the filter for signs of air leakage. Leakage may result from a worn faceplate gasket, Figure 4.8, or an improperly installed gasket. If a leakage is observed, void the sample, determine the cause and take corrective action before starting another sampling period. Generally a gasket deteriorates slowly, and the operator can decide well in advance (by increasing fuzziness of the sample outline) to change the gasket before a total failure results.

2. Visually inspect the gasket face to see if glass fibers from the filter are being left behind, resulting from over-tightening of the faceplate wing-nuts and cutting the filter along the gasket interface.

3. The operator should check the exposed filter for physical damage that may have occurred during sampling or after sampling. Physical damage to the filter after sampling does not always invalidate the sample as long as all pieces of the fliter are included in the folder. However, any loss of sample due to leakages during the sampling period or to loss of loose particulates from the filter after sampling (e.g., loss of particulates when folding the filter) invalidates the sample. The operator should mark all such samples void and forward them to the laboratory.

4. The appearance of the particulates should be checked. Any changes from the normal color, for example, may indicate new emission sources or construction activity in the area. The change should be noted on the filter folder along with any obvious reasons for the change.

4.5 Documentation

The following listed information must be recorded on the filter folder or on a field data sheet by the persons indicated.

1. By the operator who starts the sample:
    a. Station location.
    b. Project.
    c. Site number.
    d. Sampler ID number.

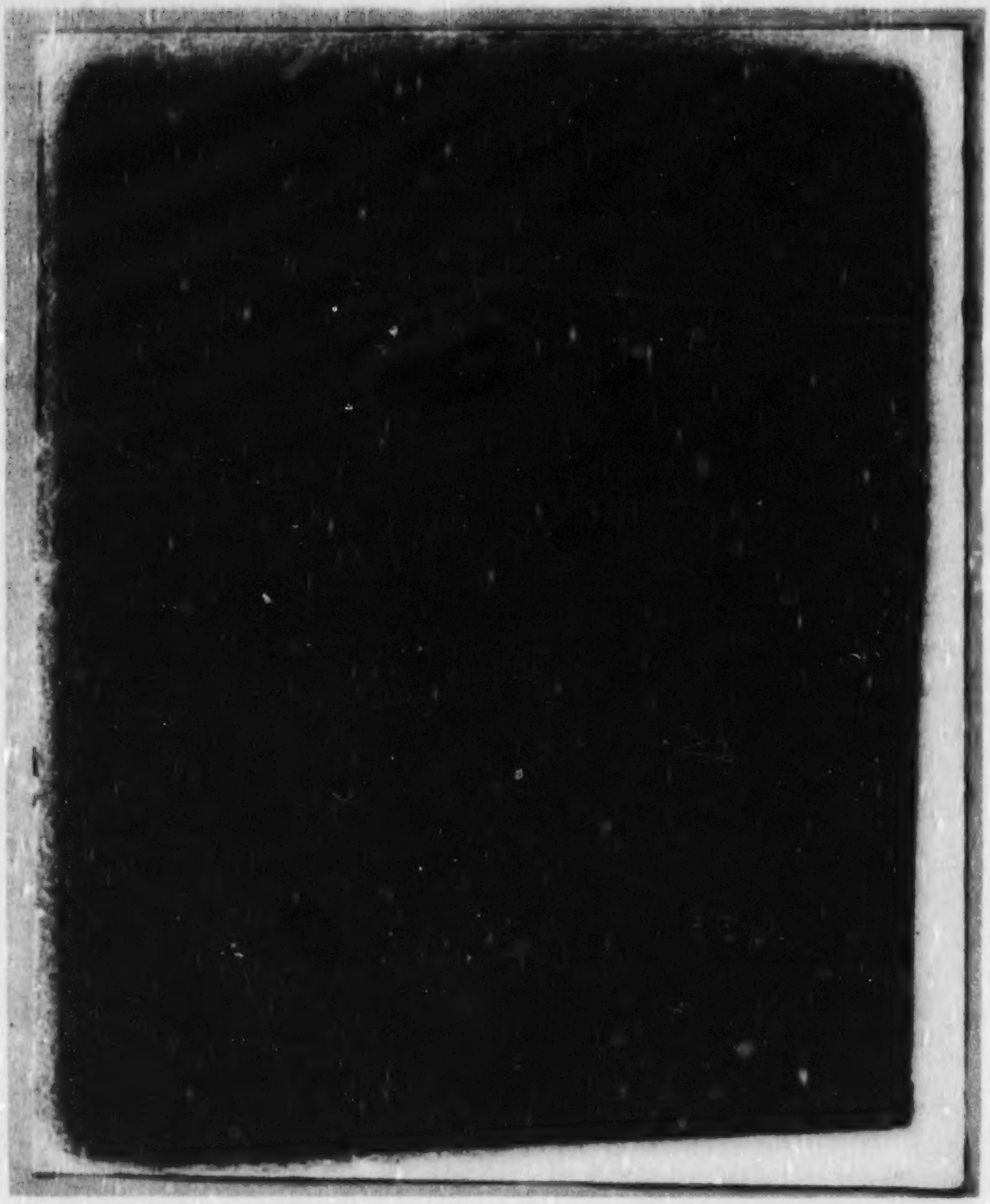


Figure 4-8. Example of air leakage around filter due to worn faceplate gasket or improper installation.

- e. Filter number.
- f. Starting time.
- g. Initial flow rate (if using rotameter).
- h. Summary of any unusual condition that may affect results (e.g., subjective evaluation of the pollution that day, construction activity, meteorology, etc.).
- i. Signature.

2. By the operator who removes the sample:
   - a. Stop time, and if available, elapsed time.
   - b. Final flow rate (or flow-rate chart must accompany the sample).
   - c. Summary of existing conditions that may affect results.
   - d. Signature.
3. By the person who transfers the sample to the laboratory:
   - a. Receiving date and his initials.
   - b. Shipping date and his initials.

Table 4.1 ACTIVITY MATRIX FOR SAMPLING PROCEDURE

Activity	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Filter installation	Filter rough side up, centered on screen, edges parallel to edges of screen and to faceplate gasket. Gasket tightened to prevent leakage.	Visually check each filter	Void filter, install substitute filter
Operational checks	1. Flow rate: stabilized initial flow rate = established initial flow rate ± 10% 2. Sampling time: 23-25 hours	1. Visually check each filter 2. Timing device	1.Perform listed steps in order until flow rate is within limits or cause of discrepancy is identified -measure line voltage -change filter -check calibration -calibrate sampler 2.Void sample
Final flow rate check	Greater than 1.14 m^3/min (40 ft^3/min)	Check each sample	Void sample
Sample handling	Absence of evidence of malfunction in post-sampling check	Visually check each sample	Void, correct cause of malfunction
Documentation	Pertinent names, sampling dates, times, sample no., filter no., station no., unusual conditions, flow rates, handling dates. (Above to be recorded on sample envelope).	Visually check each sample envelope	Complete or correct documentation if unavailable, void sample

5.0 ANALYSIS OF SAMPLES

An activity matrix summarizing the major quality control related functions for sample analyses is presented in Table 5.1 at the end of this section.

5.1 Verify Documentation and Inspect Sample

1. Upon receipt of the sample from the field, remove the filter folder from its shipping envelope and examine the hi vol field data sheet, Figure 3.2, to determine whether all data necessary to verify the sample for analysis and to calculate concentration have been provided. Void the sample if: (1) data are missing and unobtainable upon inquiry to the field operator, and (2) sampler malfunction is evident, e.g., obvious faceplate gasket leakage.

2. Record the filter number on the hi vol field data sheet and on the laboratory data log, Figure 3.1.

3. Examine the shipping envelope for sample material that may have become dislodged from the filter. If such material is observed, recover as much as possible by brushing it from the envelope to the deposit on the filter with a soft camel hair brush.

4. Examine the filter for insects embedded in the sample deposit and if found, remove them with Teflon tipped tweezers, using care to avoid disturbing more of the sample deposit than is necessary. If more than 10 insects are observed refer the sample to the supervisor for a determination whether to accept or reject it.

5. Record data verification, sample inspection, and removal of insects under Remarks in the laboratory data log, Figure 3.1.

5.2 Filter Equilibration

Equilibrate exposed filters in a conditioning environment for 24 hours, additional equilibration time up to 48 hours may be necessary for very damp filters. An equilibration chamber, employing a saturated chemical solution that will result in a relative humidity of less than 50 percent at a temperature of approximately 20 to 25°C is recommended in lieu of a controlled temperature/humidity weighing room.

An air conditioned room may be used for equilibration. A relative humidity less than 50 percent and constant within ±5 percent, and an air temperature constant within ±3°C must be maintained in the room while filters are equilibrating. A conveniently working relative humidity is 40 percent. A hygrometer is maintained in the room and the relative humidity must be checked daily.

Record hygrometer readings and any equilibration chamber malfunction, discrepancies, or maintenance in the laboratory data log, Figure 3.1.

5.3 Gravimetric Analysis

Perform a balance check as specified in Section 2.1. Weigh exposed filters to the nearest milligram on the analytical balance within 30 seconds after removal from the equilibration chamber. The weighing location should be in the conditioning environment if practicable, otherwise the analytical balance should be as close as possible to the conditioning chamber in a location that is relatively free of air currents and is at or near the temperature of the chamber. Record the weight in the laboratory data log and on the hi vol field data sheet.

Table 5.1 ACTIVITY MATRIX FOR ANALYSIS OF SAMPLES

Activity	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Verify documentation and inspect sample	-Complete documentation -Absence of evidence of malfunction or sample loss; ten or fewer insects visible in sample	Visual check	Void sample
Filter equilibration	Equilibration in controlled environment for no less than 24 hours. Room: Relative humidity < 50% and within ± 5%. Temperature constant within ± 3°C. Constant humidity chamber employing a saturated chemical solution to afford an RH< 50%, temperature between 20 and 25°C	For each sample observe: -Room or chamber conditions -Equilibration period	Repeat equilibration for 24 hours at controlled conditions
Gravimetric analysis	Determine indicated weight to nearest milligram within 30 seconds after removal from equilibration environment	Conduct gravimetric test weighing	• Report to supervisor • Reweight after equilibration for 24 hours at controlled conditions

6.0 CALCULATIONS AND DATA REPORTING

An activity matrix summarizing the quality control functions for the calculations and data reporting requirements is presented in Table 6.1.

6.1 Sample Air Volume

1. For samplers equipped with rotameters:

$$V = \left(\frac{Q_i + Q_f}{2}\right) t$$

where V = air volume sampled, m^3
Q_i = initial air flow rate, m^3/min at STP
Q_f = final air flow rate, m^3/min at STP
t = sampling period (elapsed time), min.

2. For samplers equipped with flow recorders:

$$V = Q\ t$$

where V = air volume sampled, m^3
Q = average sampling rate, m^3/min at STP
t = sampling period (elapsed time), min

The average sampling rate, Q, is determined from the recorder chart by estimation. If the flow rate does not vary more than 0.11 m^3/min during the sampling period, read the flow rate from the chart at 2-hour intervals and take the average for the value Q.

6.1.1 Sample Concentration

$$S.P. = \frac{(W_f - W_i)\ 10^6}{V}$$

where S.P. = concentration of suspended particulate, $\mu g/m^3$
W_f = weight of exposed filter, grams
W_i = tare weight of filter, grams
V = air volume sampled, m^3

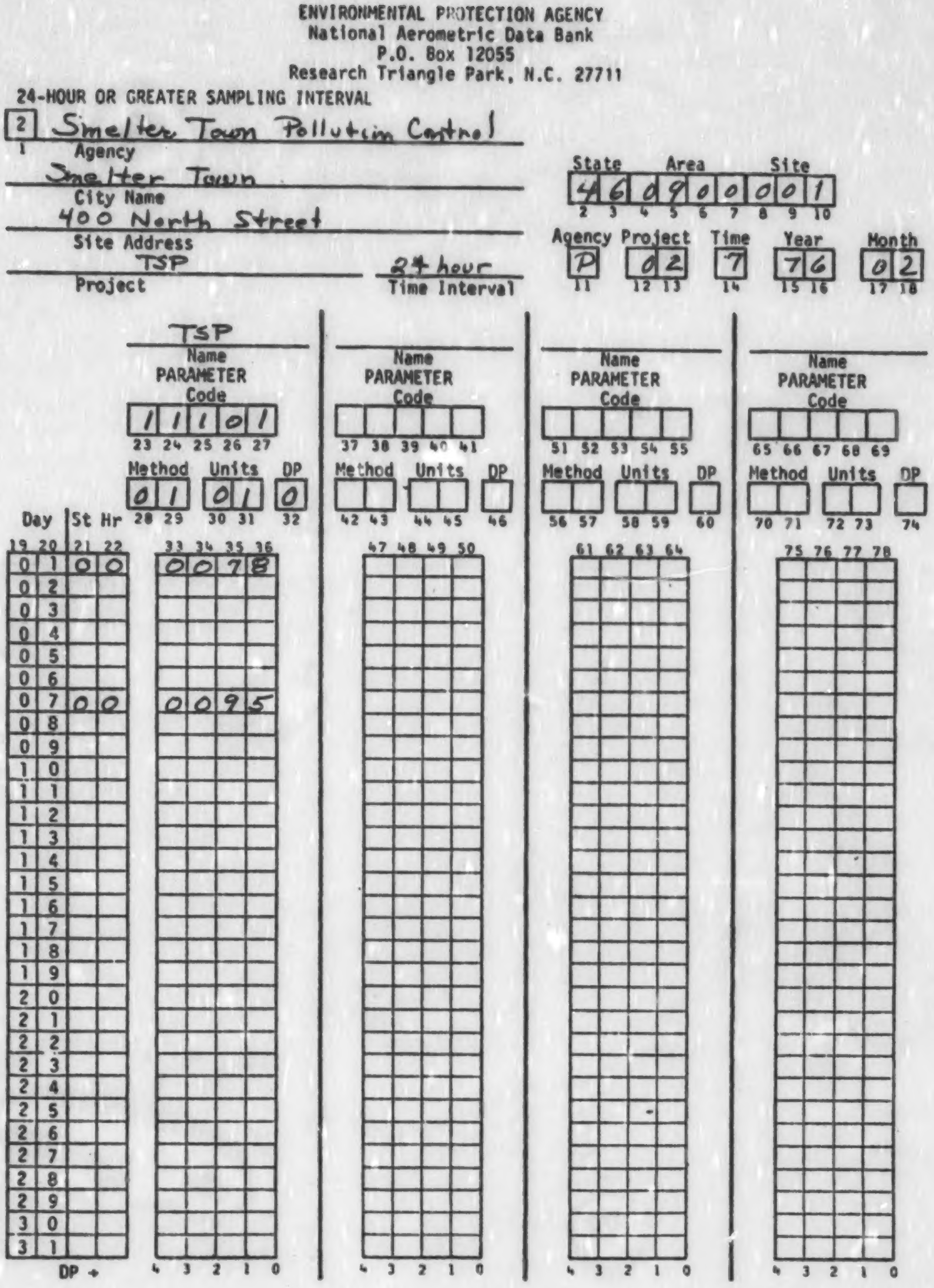
Record all original calculations in the laboratory data log, Figure 3.1.

6.2 Document and Report Data

Record in micrograms per cubic meter, all daily concentration levels with required identifying information on the SAROAD Daily Data Form, Figure 6.1. See AEROS User's Manual, OAQPS No. 1.2-039, for detailed instructions.

Table 6.1 ACTIVITY MATRIX FOR CALCULATIONS AND DATA REPORTING

Activity	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Calculations (1) Sample volume (2) Sample concentration	All needed data available	Visual check for each sample	Void sample
Documentation and sample verification	-Documentation complete for calculation of concentration -All sample and data identification numbers match -Absence of evidence of malfunction or sample loss	Visual check for each sample	Void sample
Document and report data	All needed data available	Visual check for each sample	Void sample



ENVIRONMENTAL PROTECTION AGENCY
National Aerometric Data Bank
P.O. Box 12055
Research Triangle Park, N.C. 27711

24-HOUR OR GREATER SAMPLING INTERVAL

2 (1) Agency: Smelter Town Pollution Control

City Name: Smelter Town

Site Address: 400 North Street

Project: TSP

Time Interval: 24 hour

State (2 3): 4 6 — Area (4 5 6 7): 0 9 0 0 — Site (8 9 10): 0 0 1

Agency (11): P — Project (12 13): 0 2 — Time (14): 7 — Year (15 16): 7 6 — Month (17 18): 0 2

Name PARAMETER	TSP			
Code	1 1 1 0 1 (23 24 25 26 27)	(37 38 39 40 41)	(51 52 53 54 55)	(65 66 67 68 69)
Method	0 1 (28 29)	(42 43)	(56 57)	(70 71)
Units	0 1 (30 31)	(44 45)	(58 59)	(72 73)
DP	0 (32)	(46)	(60)	(74)

Day 19 20	St Hr 21 22	33 34 35 36	47 48 49 50	61 62 63 64	75 76 77 78
0 1	0 0	0 0 7 8			
0 2					
0 3					
0 4					
0 5					
0 6					
0 7	0 0	0 0 9 5			
0 8					
0 9					
1 0					
1 1					
1 2					
1 3					
1 4					
1 5					
1 6					
1 7					
1 8					
1 9					
2 0					
2 1					
2 2					
2 3					
2 4					
2 5					
2 6					
2 7					
2 8					
2 9					
3 0					
3 1					
DP →		4 3 2 1 0	4 3 2 1 0	4 3 2 1 0	4 3 2 1 0

Figure 6.1 SAROAD daily data form

7.0 MAINTENANCE

Scheduled or preventive maintenance of the sampling equipment will result in a reduction of downtime and remedial maintenance requirements. Since the sampling equipment is only operated on a intermittent basis, the frequency of maintenance is a function of the actual number of hours of use. Normally two to three remedial maintenance activities are required per year. Table 7.1 at the end of this section is an activity matrix summarizing the quality assurance aspects of major maintenance checks. Record all maintenance activities in a maintenance log book. Maintenance can be best performed within the laboratory facility and not in the field. Often spare motors, on which maintenance has been performed, are carried to the field for installation and then calibration.

7.1 Sampler Motor

Motor brushes usually require replacement after 400 to 500 hours of operation at normal line voltage (115 V). The brushes should be replaced before they are worn to the point that motor damage can occur. The optimum replacement interval must be determined from experience. Manufacturer's instructions should be followed in replacing the brushes. Recalibration of the rotameter is necessary after brushes are replaced. The recalibration should not, however, be performed on a motor without an initial break-in period to properly seat the brushes against the armature. This period usually requires running the sampler against a resistance equivalent to a clean filter or a No. 18 calibration plate for a period of several hours. A flow diagram is presented in Figure 7.1 describing the various steps required for motor maintenance.

Record all sampler maintenance operations with date performed and operator's initials in the sampler log book and on a label which is to be attached to the sampler motor.

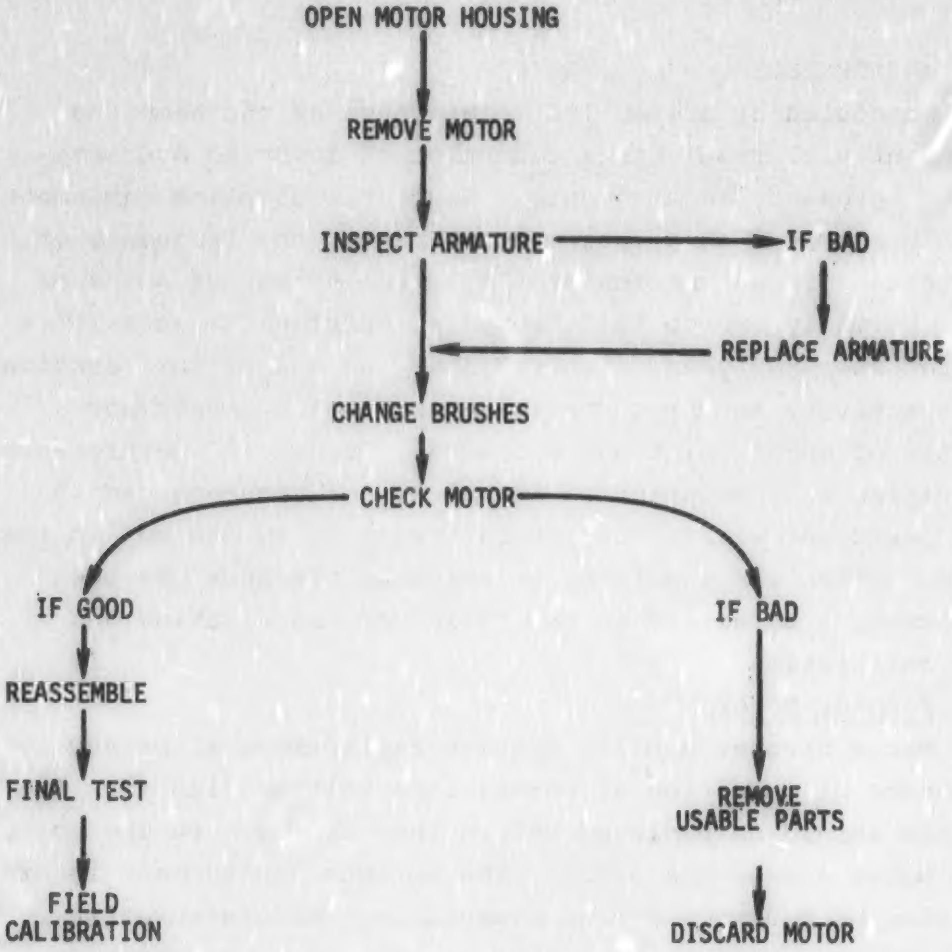

OPEN MOTOR HOUSING
REMOVE MOTOR
INSPECT ARMATURE
IF BAD
REPLACE ARMATURE
CHANGE BRUSHES
CHECK MOTOR
IF GOOD
IF BAD
REASSEMBLE
FINAL TEST
FIELD CALIBRATION
REMOVE USABLE PARTS
DISCARD MOTOR

Figure 7.1 Flow diagram for hi vol motor maintenance

An example of this gummed label is represented in Figure 7.2.

7.2 Faceplate Gasket

A worn faceplate gasket is characterized by a gradual blending of the interface between collected particulates and the clean filter border. Any decrease in the sharpness of this interface indicates the need for a new faceplate gasket.

The old gasket can be removed with a knife and the surface properly cleaned. A new gasket is then sealed to the faceplate with rubber cement or double-sided adhesive tape.

Record all gasket replacements with date and operator's initials in the sampler log book.

7.3 Rotameter

Clean and calibrate the rotameter of a sampler when the float behaves in an erratic manner, or when moisture or foreign matter is detected in the rotameter.

Clean the rotameter prior to routine calibration (alcohol is a satisfactory cleaning solvent). A flow diagram showing the required maintenance steps is presented in Figure 7.3.

HI VOL MOTOR NO:____________________

SITE LOCATION:____________________

LAST MAINTENANCE:____________________

LAST CALIBRATION:____________________

CHECKED BY:____________________

NEXT MAINTENANCE DUE:____________________

NEXT CALIBRATION DUE:____________________

Figure 7.2 Example of label for sampler motor

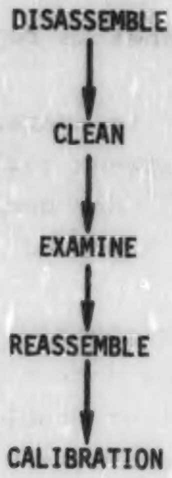


Figure 7.3 Maintenance requirements for rotameter service

7.4 Sampling Head

Leaks in the sampling head are not experienced frequently. The welded seams and the condition of the guide pins on the top surface of the head should be checked initially by visual inspection. Should a defect be suspected, assemble the sampling head to the motor, install a filter for resistance, and apply a soap solution to the suspect problem area. Disassemble the sampling head and examine the inside of the head for the presence of soap solution. If the soap solution is present inside the sampling head a leak is present. Repair or discard the sampler head.

7.5 Motor Gaskets

Two gaskets are employed with each sampler. The top gasket is approximately 3/16-inch thick rubber and the bottom gasket is approximately 3/4-inch thick foam rubber. Both should be inspected for wear and replaced if necessary.

7.6 Flow Transducer and Recorder

There is no routine maintenance required with respect to this device. Should a malfunction occur, it should be replaced with a new recorder.

Table 7.1 ACTIVITY MATRIX FOR MAINTENANCE

Equipment	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Sampler motor	-400 hours operation of motor brushes -Absence of malfunction	Visually check upon receipt and after each 400 hours of operation	-Replace motor brushes -Other maintenance as indicated
Faceplate gasket	Absence of leaks at filter seal	Visually check after each sampling period	Replace gasket
Rotameter	-Absence of foreign materials -Stable operations	Visually check for each sample	Clean; replace if damaged
Motor gaskets	Leak tight fit	Visually check each 400 hours of operation	Replace gaskets
Sampling head	Absence of leaks	Visually check each 200 hours of operation	Replace sampling head

8.0 AUDITING PROCEDURE

Audit, as used here, implies an independent assessment of the quality of data obtained by the ambient air monitoring methods. Independence can be achieved by having the audit made by a different operator/analyst than the one conducting the routine field measurements. The audit should be a true assessment of the measurement process under normal operation, i.e., without any special preparation or adjustment of the system. Routine quality assurance checks conducted by the operator/analyst are necessary for obtaining and reporting good quality data, but they are not to be considered as part of the auditing procedure.

Two types of audits are recommended herein, performance and system audits. Four performance audits and a systems audit are described in detail in the following sections. An alternative to performing four individual audits is to audit the entire measurement process by comparing the final results from the field sampler to those obtained with a collocated sampler. This alternative is described in Section 8.2. A summary of these audits is given in Table 8.2 at the end of this section. A systems audit is described in Section 8.3.

Proper implementation and conduct of an auditing program will serve a two-fold purpose: (1) to screen the data, by lots of say N = 50 to 100 measurements of concentration of TSP to detect whether the data quality is adequate, and (2) to estimate data quality in terms of the precision and accuracy. Appendices G and I in Volume I of this Handbook[4] contain information for the auditing and sampling procedures presented in this section and techniques for estimating precision and accuracy of the data.

8.1 Performance Audits

Performance audits are independent assessments made by another operator/analyst to evaluate the quality of data produced by the total measurement system (sample collection, sample analysis, and data processing). Performance audits are normally a quantitative appraisal of quality.

Four performance audits of individual variables are recommended:

1. Audit of clean filter weighing.
2. Audit of flow rate calibration.
3. Audit of exposed filter weighing.
4. Audit of data processing.

An auditing level of 7 out of 100 sampling periods is suggested here as a starting frequency. For the case where one sample is collected every sixth day, an auditing level of one per month is recommended. This would result in an auditing level of approximately 3 for a lot size of 15 for data reported quarterly. If the number of sampling periods is greater than 15 but less than 50, four (4) audits are recommended. These frequencies are suggested starting frequencies and they are to be altered based on experience and data quality. The audit frequency should be reduced if past experience indicates the data are of good quality, or increased if data are of poor quality. In determining the number of audits to be made for a large lot size, it is more important to make sure that the sample is representative of the various conditions that may influence data quality than to adhere to a fixed frequency. The supervisor/quality assurance coordinator will specify the audits and auditing level to be used according to monitoring requirements.

8.1.1 Clean Filter Weighing Audits - Weighing audits are made as soon as practical before or after the regular weighing. Clean filters are normally weighed in batches. This allows for the sampling to be performed and corrections to be made before the filters are used.

1. Divide into lot sizes of 100 or less and weigh.
2. Randomly select and reweigh 7 filters from each lot of 100. See Appendix I of Volume I of this Handbook for

recommending sampling procedures.[4]

3. If any one of the 7 check weights differs more than 2.8 mg from the original weight, reweigh all the filters in that lot. Record results in a laboratory log book.

8.1.2 Flow Rate Calibration Audits - Independent flow rate calibration audits should be made on site. Portable audit equipment is used.[a] Perform the flow rate calibration audits according to the following procedure:

1. Set up equipment.
2. Select one of the resistance plates and obtain the actual flow rate, Q_a, and the rotameter reading, following the calibration procedures, Section 2.6.
3. Convert rotameter reading to flow rate, Q_m, using the calibration curve and making corrections for ambient temperature and pressure.
4. Compute the percent accuracy

$$\% A = \frac{Q_m - Q_a}{Q_a} \times 100. \qquad \text{Equation 8-1}$$

5. If the percent accuracy is greater than ± 7 percent for any one check, a complete recalibration should be performed before sampling is resumed.
6. Report Q_m, Q_a, and the percent accuracy, % A, on an $\overline{X}$ and R chart, under measurement result, items 1 and 2. Record % A in the cells preceded by "Range, R" as indicated in Figure 8.1. The value, % A, can be positive or negative and the range is always positive. The sign of the difference should be retained to determine the existence of trends and/or consistent biases. The steps involved in the construction of a quality control chart and in the interpretation of the results are described in Appendix H, Volume I of this Handbook.[4]

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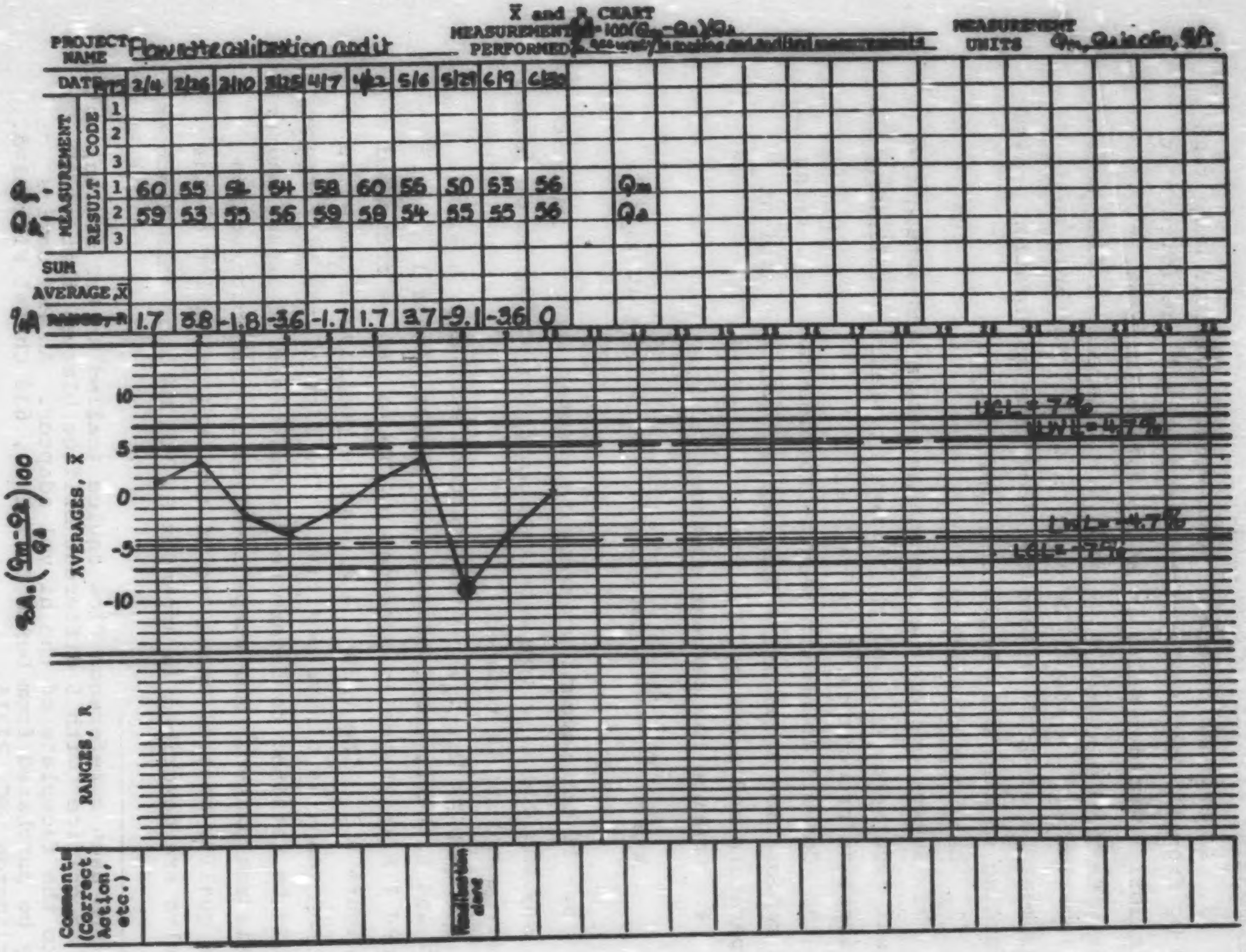
[a] US EPA uses a reference flow device (called an ReF device) which is an orifice (with 5 different orifice plates) that mount onto the faceplate of the hi vol adaptor. An ReF device may be purchased from Dexco Co., Inc., 630 Chapel Hill Blvd., Burlington, NC 27215.



$\bar{X}$ and R CHART

PROJECT NAME Flow rate calibration audit

MEASUREMENT PERFORMED %A = 100($Q_m - Q_a$)/Q_a [illegible]

MEASUREMENT UNITS Q_m, Q_a in cfm, [illegible]

DATE	2/4	2/26	3/10	3/25	4/7	4/22	5/6	5/29	6/19	6/30	
MEASUREMENT CODE 1											
MEASUREMENT CODE 2											
MEASUREMENT CODE 3											
Q_m – MEASUREMENT RESULT 1	60	55	54	54	58	60	56	50	53	56	Q_m
Q_a – MEASUREMENT RESULT 2	59	53	55	56	59	58	54	55	55	56	Q_a
MEASUREMENT RESULT 3											
SUM											
AVERAGE, $\bar{X}$											
%A ~~RANGE, R~~	1.7	3.8	-1.8	-3.6	-1.7	1.7	3.7	-9.1	-3.6	0	

%A = $\left(\frac{Q_m - Q_a}{Q_a}\right)100$
AVERAGES, $\bar{X}$
10
5
0
-5
-10
UCL = 7%
UWL = 4.7%
LWL = -4.7%
LCL = -7%
RANGES, R
Comments (Correct Action, etc.)

Figure 8.1 Quality control chart for flow rate calibration audit

7. Repeat the above for each flow rate calibration audit plotting all points on the chart and connecting the points by a straight line.

8. Tentative warning and control lines are given as ± 4.7 percent (warning lines) and ± 7 percent ("out of control" lines). After 15 to 20 points are plotted, new control and warning lines may be derived as described in Appendix H of Volume I of this Handbook.[4]

9. Out of control points are an indication of calibration errors, instrument damage, etc. Recalibrate the sampler prior to further sampling when out of control.

10. Forward the $\overline{X}$ and R chart to the supervisor for review.

8.1.3 **Exposed Filter Weighing Audits** - In order to allow for possible data corrections, it is necessary to weigh exposed filters immediately after a 24-hour conditioning period. Thus, it may be impossible to have lot sizes greater than 10 or 20.

1. Randomly select and reweigh 4 out of every lot size of 50 or less (this would mean 100 percent checking if 4 or less exposed filters are weighed at one time). For lot sizes of 50 or greater, reweigh 7 from each lot.

2. Reweigh all filters in a lot if any audit weight differs by more than ± 5.0 mg from the original weight.

3. Accept the lot with no change if all audits are within ± 5.0 mg of the originals.

4. Record the original and audit weights on an $\overline{X}$ and R chart as illustrated in Figure 8.2. Follow the procedure outlined in steps 6 through 10 in Section 8.1.2. The accuracy A, in this case would be defined as

$$A = \text{Original Weight} - \text{Audit Weight (mg)}. \qquad \text{Equation 8-2}$$

Tentative warning and control limits of ± 3.3 mg and ± 5.0 mg, respectively, are recommended until sufficient data are obtained

$\bar{X}$ and R CHART

PROJECT NAME Audits of weights of exposed filters

MEASUREMENT PERFORMED A = routinely measured weight - audited weight (mg)

MEASUREMENT UNITS mg

DATE '76	1/1	1/8	1/15	1/22	1/29	2/5	2/12	2/19	2/26	3/4	3/11	3/18	3/25	4/1	4/8	4/15	4/22	4/29	5/6	5/13	5/20	5/27	6/3	6/10	
MEASUREMENT CODE 1	Lot 1								Lot 2								Lot 3								
MEASUREMENT CODE 2																									
MEASUREMENT CODE 3																									
Original weight — MEASUREMENT RESULT 1	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
Audit weight — MEASUREMENT RESULT 2	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
MEASUREMENT RESULT 3																									
SUM																									
AVERAGE, $\bar{X}$																									
A ~~RANGE, R~~	3.2	3.8	-3.8	3.7	1.8	4.4	2.9	2.7	-2.1	1.6	-5.4	3.6	-4.4	4.0	4.9	-3.9	4.3	-2.8	1.3	3.7	4.5	-1.5	4.2	-3.1	

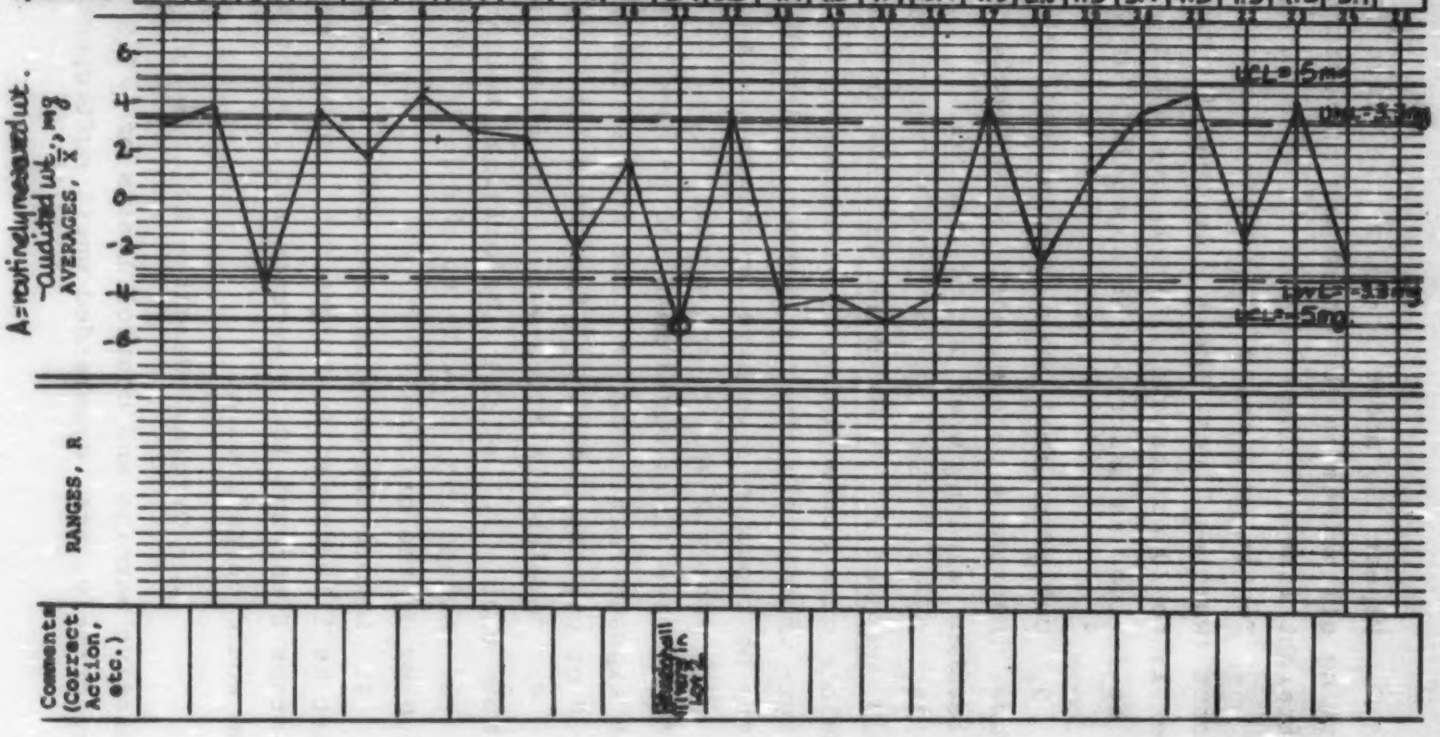


Figure 8.2 Quality control chart for audit of weights of exposed filters

to support an alteration of these limits. Do not increase the limits unless so authorized by the Administrator.

5. Forward the $\overline{X}$ and R chart to the supervisor for review.

6. Out of control points indicate the need for recalibration of the balance and/or improved operator technique. Reweigh all of the remaining exposed filters in the lot.

8.1.4 Data Processing Check - In auditing data processing procedures, it is convenient and allows for correction to be made immediately if audits are made soon after the original calculations have been performed. In particular, this allows for possible retrieval of additional explanatory data from field personnel when necessary.

1. Use the same audit rate as step 1 of 8.1.3.

2. The check is made starting with the raw data on the data sheet or flow rate recorder chart and continuing through the recording of the concentration in $\mu g/m^3$ on the SAROAD form.

3. If the mass concentration of suspended particulates computed by the audit check, $(\mu g\ TSP/m^3)_a$, differs from the original value, $(\mu g\ TSP/m^3)_m$ by as much as ± 3 percent, all samples in that lot are checked and corrected as necessary. The audit value is always given as the correct value under the assumption that a discrepancy between the two values is always double checked by the auditor.

4. Audit values are recorded in the data log and reported, along with the original values, to the supervisor for review.

8.2 Auditing with a Collocated Sampler

An alternate method of auditing the high volume method, which in certain situations might be feasible, is to use two collocated samplers. A network operating several samplers in a reasonably small area (e.g., city or county) might find this method more convenient than auditing individual variables. The field sampler and the audit sampler will be denoted respectively, as the first and second sampler throughout the section.

The second sampler should be operated in strict accordance with the procedures given in this section of the Handbook. A record should be maintained of the checks performed on the second sampler and reported with the data as requested by the manager. An audit would be to place the second sampler adjacent to (but no closer than 6 feet) the field sampler (see Reference 1 for discussion on positioning the sampler) and sample simultaneously.

The percent difference in the concentration of suspended particulates as measured by the field sampler and the second sampler is computed by

$$\text{Percent difference} = \%D = \frac{(\mu\text{gTSP/m}^3)_1 - (\mu\text{gTSP/m}^3)_2}{0.5\left[(\mu\text{gTSP/m}^3)_1 + (\mu\text{gTSP/m}^3)_2\right]} \times 100. \quad \text{Equation 8-3}$$

Based on the results of a collaborative test[1] a defect would be defined as

$$|D| \geq 15.^*$$

The auditing level for collocated samplers would be the same as that given in the previous section, i.e., $n = 7$, $N = 100$ as the initial rate.

Values of $(\mu\text{g TSP/m}^3)_1$, $(\mu\text{g TSP/m}^3)_2$, and D, and the auditing level would be reported on the quality control $\overline{X}$ and R chart along with standard identification information.

The data may be analyzed and reported as described in steps 6 through 10 of Section 8.1.2, above, with the exception that the difference, D, is obtained by equation 8-3. Tentative warning and control limits are given as 10.0 and 15.0 percent respectively. Out of control values may imply errors in any one of the several sample collection and analysis steps. Audits of specific steps will aid in determining the likely cause of the large deviations, e.g. calibration, weighing, and/or data processing errors. See Figure 8.3 for an example quality control chart.

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*If $\sigma = 3.5$ percent of the concentration of TSP for each sampler, then the percent difference, D, would have a standard deviation of 5.0 percent of the mean value. This gives a 3σ value of 15 percent.

8.3 Systems Audit

A systems audit is an on-site inspection and review of the quality assurance system used for the total measurement system (sample collection, sample analysis, data processing, etc.). System audits are normally a qualitative appraisal of system quality.

A systems audit should be conducted at the beginning of a new monitoring system and as appropriate thereafter to audit significant changes in system operation.

A preliminary form for use in a system audit is given in Table 8.1. These questions should be checked for the applicability to the particular local, state, or Federal agency.

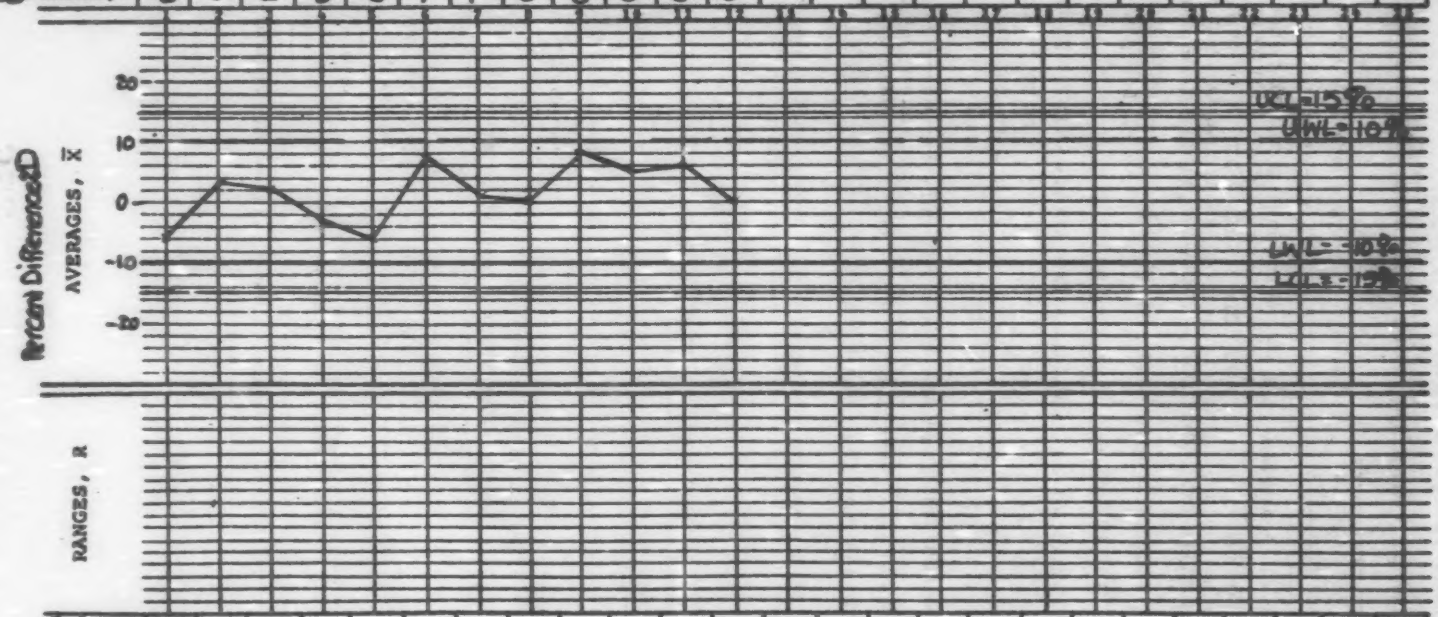
$\bar{X}$ and R CHART

PROJECT NAME Audit with collocated samplers

MEASUREMENT PERFORMED $\%D = \frac{[(\mu g\ TSP/m^3)_1 - (\mu g\ TSP/m^3)_2]}{\text{average of } (\mu g\ TSP/m^3)\ 1 \text{ and } 2}$

MEASUREMENT UNITS Percent difference

DATE			1/1	1/[illegible]	1/15	1/22	1/29	2/5	2/12	2/19	2/[illegible]	3/4	3/11	3/[illegible]												
MEASUREMENT	CODE	1	Lot	1 →			Lot	2 →			Lot	3 →														
		2																								
		3																								
	RESULT	1	172	69	99	55	148	161	95	121	142	132	154	45												
		2	182	67	96	57	158	150	94	121	131	125	145	45												
		3																								
SUM																										
AVERAGE, $\bar{X}$			177	68	97	56	153	156	94.5	121	136.5	128.5	149.5	45												
%D RANGE, R			-6	3	2	-3	-6	7	1	0	8	6	6	0												



Comments (Correct. Action, etc.)

Figure 8.3 Quality control chart for audit with collocated samplers

Table 8.1 CHECK LIST FOR USE BY AUDITOR FOR HI VOL METHOD

1. What type of hi vol samplers are utilized in the network? ______________

2. How often are the samplers run? (a) daily, (b) once every six days, (c) once every 12 days, (d) other.

3. What type of filter and number is being utilized? ______________

4. Are there any pre-exposure checks for pin holes or imperfections run on the filters? ___

5. What is the collection efficiency for your filters? ______________

6. What is the calibration procedure for the hi vol sampler? ______________

7. Which statement most closely estimates the frequency of flow rate calibration? (a) once when purchased, (b) once when purchased, then after every sampler modification, or (c) once when purchased, then at regular intervals thereafter.

8. Are flow rates measured before and after sampling period? ___

9. If the answer to #8 is yes, using the equation below, what is the estimated average percent of change in the flow rates? Use the equation

$$\frac{100(Q_i - Q_f)}{Q_i} = \text{percent change}$$

(a) less than 10%, (b) 10-20%, or (c) greater than 20%.

10. Is there a log book at each sampler to record flows and times? ___

11. Are filters conditioned before initial and final weighings? ___. If so, how long? _________. And at what percent humidity? __________.

12. Is the balance checked on a periodic basis? ___. If so, how often? With which standard weights? ______________

13. How often are the hi-vol filters weighed? ______________ How is the data from these weighings handled? ______________

14. Are all weighings and serials numbers of filters kept in a log book at the laboratory? ___

15. What is the approximate time delay between sample collection and final weighing? _____days

Table 8.2 ACTIVITY MATRIX FOR AUDITING PROCEDURE

Audit	Acceptance limits	Frequency and method of measurement	Action if requirements not met
Weighing audits Clean filters Exposed filters	Audit wt. = original wt. ± 2.8 mg Audit wt. = original wt. ± 5.0 mg	Frequency - 7 audits per 100 filters. For a lot size of 50 or less filters make 4 audits. Method - Use analytical balance. Condition filters for 24 hours before weighing	Reweigh all filters in the batch
Calibration audit	$0.93 \le \frac{\text{Flow rate}_m}{\text{Flow rate}_a} \le 1.07$ Flow rate_m = routine measured rate Flow rate_a = audited rate	Frequency - See above. Method - Same as for calibration procedure for one flow rate.	Perform calibration before sampling is resumed.
Data processing audit	$0.97 < \frac{(\mu g\ TSP/m^3)_m}{(\mu g\ TSP/m^3)_a} < 1.03$ $(\mu g\ TSP/m^3)_m$ = routine measured concentration $(\mu g\ TSP/m^3)_a$ = audited concentration	Frequency - See above. Method - Redo all calculations; readings of charts, etc. through recording results.	Recheck all calculations.
Collocated sampler audit	$\frac{100[(\mu g\ TSP/m^3)_1 - (\mu g\ TSP/m^3)_2]}{\text{Average } (\mu g\ TSP/m^3)_1 \text{ and }_2} \le \pm 15\%$, i.e., the difference in concentration does not exceed 15% of the average value obtained by the field sampler 1 and the collocated sampler 2. These limits are for repeatability (same operator). Larger limits would be expected for reproducibility (between two operators).	Frequency - See above. Method - Collocated monitor calibrated and maintained in strict accordance to method description.	Data invalid for sampling period.
Systems audit	Method as described in this section of the Handbook	Frequency - At the beginning of a new monitoring system and periodically as appropriate Method - Observation of procedures and by check list	Initiate improved methods and/or training programs

9.0 RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY

To achieve data of desired quality, two essential considerations are necessary, (1) the measurement process must be in a state of statistical control at the time of the measurement and (2) the systematic errors, when combined with the random variation (errors or measurement), must result in a suitably small uncertainty.[5] As evidence in support of good quality data it is necessary to perform quality control checks and independent audits of the measurement process, to document these data by means of a quality control chart, for example, as described in Section 8 for audits, and to use materials, instruments, and measurement procedures that can be traced to an appropriate standard of reference.

Data must be routinely obtained by repeat measurements of standard reference samples, primary, secondary and/or working standards and by the establishment of a condition of process control. The working calibration standards should be traceable to standards of higher accuracy, such as those listed in Table 9.1.

Table 9.1 LIST OF RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY

1. Class "S" weights (made to NBS specifications) are recommended for the analytical balance calibration. See Section 2.1 for details on balance calibration checks.

2. Positive displacement roots meter is recommended for calibrating the orifice used to calibrate the high volume sampler rotameter. See Section 2.5 for details on high volume sampler rotameter calibration.

3. Positive displacement roots meter is recommended for calibrating the device (including resistance plate) used to audit the high volume sampler flow rate calibration. See Section 8.1.2 for details on flow rate calibration audits.

4. The elapsed time meter, checked against an accurate timepiece on a semi-annual basis, must be within ± 2 minutes per day.

10.0 REFERENCE METHOD*

Appendix B—Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method)

1. *Principle and Applicability.*

1.1 Air is drawn into a covered housing and through a filter by means of a high-flow-rate blower at a flow rate (1.13 to 1.70 m.^{3}/min.; 40 to 60 ft.^{3}/min.) that allows suspended particles having diameters of less than 100 μm. (Stokes equivalent diameter) to pass to the filter surface. (*1*) Particles within the size range of 100 to 0.1μm. diameter are ordinarily collected on glass fiber filters. The mass concentration of suspended particulates in the ambient air (μg./m.^{3}) is computed by measuring the mass of collected particulates and the volume of air sampled.

1.2 This method is applicable to measurement of the mass concentration of suspended particulates in ambient air. The size of the sample collected is usually adequate for other analyses.

2. *Range and Sensitivity.*

2.1 When the sampler is operated at an average flow rate of 1.70 m.^{3}/min. (60 ft.^{3}/min.) for 24 hours, an adequate sample will be obtained even in an atmosphere having concentrations of suspended particulates as low as 1 μg./m.^{3}. If particulate levels are unusually high, a satisfactory sample may be obtained in 6 to 8 hours or less. For determination of average concentrations of suspended particulates in ambient air, a standard sampling period of 24 hours is recommended.

2.2 Weights are determined to the nearest milligram, airflow rates are determined to the nearest 0.03 m.^{3}/min. (1.0 ft.^{3}/min.), times are determined to the nearest 2 minutes, and mass concentrations are reported to the nearest microgram per cubic meter.

3. *Interferences.*

3.1 Particulate matter that is oily, such as photochemical smog or wood smoke, may block the filter and cause a rapid drop in airflow at a nonuniform rate. Dense fog or high humidity can cause the filter to become too wet and severely reduce the airflow through the filter.

3.2 Glass-fiber filters are comparatively insensitive to changes in relative humidity, but collected particulates can be hygroscopic. (*2*)

4. *Precision, Accuracy, and Stability.*

4.1 Based upon collaborative testing, the relative standard deviation (coefficient of variation) for single analyst variation (repeatability of the method) is 3.0 percent. The corresponding value for multilaboratory variation (reproducibility of the method) is 3.7 percent. (*3*)

4.2 The accuracy with which the sampler measures the true average concentration depends upon the constancy of the airflow rate through the sampler. The airflow rate is affected by the concentration and the nature of the dust in the atmosphere. Under these conditions the error in the measured average concentration may be in excess of ±50 percent of the true average concentration, depending on the amount of reduction of airflow rate and on the variation of the mass concentration of dust with time during the 24-hour sampling period. (*4*)

5. *Apparatus.*

5.1 *Sampling.*

5.1.1 *Sampler.* The sampler consists of three units: (1) the faceplate and gasket, (2) the filter adapter assembly, and (3) the motor unit. Figure B1 shows an exploded view of these parts, their relationship to each other, and how they are assembled. The sampler must be capable of passing environmental air through a 406.5 cm.^{2} (63 in.^{2}) portion of a clean 20.3 by 25.4 cm. (8- by 10-in.) glass-fiber filter at a rate of at least 1.70 m.^{3}/min. (60 ft.^{3}/min.). The motor must be capable of continuous operation for 24-hour periods with input voltages ranging from 110 to 120 volts, 50–60 cycles alternating current and must have third-wire safety ground. The housing for the motor unit may be of any convenient construction so long as the unit remains airtight and leak-free. The life of the sampler motor can be extended by lowering the voltage by about 10 percent with a small "buck or boost" transformer between the sampler and power outlet.

5.1.2 *Sampler Shelter.* It is important that the sampler be properly installed in a suitable shelter. The shelter is subjected to extremes of temperature, humidity, and all types of air pollutants. For these reasons the materials of the shelter must be chosen carefully. Properly painted exterior plywood or heavy gauge aluminum serve well. The sampler must be mounted vertically in the shelter so that the glass-fiber filter is parallel with the ground. The shelter must be provided with a roof so that the filter is protected from precipitation and debris. The internal arrangement and configuration of a suitable shelter with a gable roof are shown in Figure B2. The clearance area between the main housing and the roof at its closest point should be 580.5±193.5 cm.^{2} (90±30 in.^{2}). The main housing should be rectangular, with dimensions of about 29 by 36 cm. (11½ by 14 in.).

5.1.3 *Rotameter.* Marked in arbitrary units, frequently 0 to 70, and capable of being calibrated. Other devices of at least comparable accuracy may be used.

5.1.4 *Orifice Calibration Unit.* Consisting of a metal tube 7.6 cm. (3 in.) ID and 15.9 cm. (6¼ in.) long with a static pressure tap 5.1 cm. (2 in.) from one end. See Figure B3. The tube end nearest the pressure tap is flanged to about 10.8 cm. (4¼ in.) OD with a male thread of the same size as the inlet end of the high-volume air sampler. A single metal plate 9.2 cm. (3⅝ in.) in diameter and 0.24 cm. ($\frac{3}{32}$ in.) thick with a central orifice 2.9 cm. (1⅛ in.) in diameter is held in place at the air inlet end with a female threaded ring. The other end of the tube is flanged to

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* Reproduced from Code of Federal Regulation 40, Part 50.11, Appendix B, July 1, 1975, Pages 12-16.

hold a loose female threaded coupling, which screws onto the inlet of the sampler. An 18-hole metal plate, an integral part of the unit, is positioned between the orifice and sampler to simulate the resistance of a clean glass-fiber filter. An orifice calibration unit is shown in Figure B3.

5.1.5 *Differential Manometer*. Capable of measuring to at least 40 cm. (16 in.) of water.

5.1.6 *Positive Displacement Meter*. Calibrated in cubic meters or cubic feet, to be used as a primary standard.

5.1.7 *Barometer*. Capable of measuring atmospheric pressure to the nearest mm.

5.2 *Analysis*.

5.2.1 *Filter Conditioning Environment*. Balance room or desiccator maintained at 15° to 35°C. and less than 50 percent relative humidity.

5.2.2 *Analytical Balance*. Equipped with a weighing chamber designed to handle unfolded 20.3 by 25.4 cm. (8- by 10-in.) filters and having a sensitivity of 0.1 mg.

5.2.3 *Light Source*. Frequently a table of the type used to view X-ray films.

5.2.4 *Numbering Device*. Capable of printing identification numbers on the filters.

6. *Reagents*.

6.1 *Filter Media*. Glass-fiber filters having a collection efficiency of at least 99 percent for particles of 0.3 μm. diameter, as measured by the DOP test, are suitable for the quantitative measurement of concentrations of suspended particulates, (*5*) although some other medium, such as paper, may be desirable for some analyses. If a more detailed analysis is contemplated, care must be exercised to use filters that contain low background concentrations of the pollutant being investigated. Careful quality control is required to determine background values of these pollutants.

7. *Procedure*.

7.1 *Sampling*.

7.1.1 *Filter Preparation*. Expose each filter to the light source and inspect for pinholes, particles, or other imperfections. Filters with visible imperfections should not be used. A small brush is useful for removing particles. Equilibrate the filters in the filter conditioning environment for 24 hours. Weigh the filters to the nearest milligram; record tare weight and filter identification number. Do not bend or fold the filter before collection of the sample.

7.1.2 *Sample Collection*. Open the shelter, loosen the wing nuts, and remove the faceplate from the filter holder. Install a numbered, preweighed, glass-fiber filter in position (rough side up), replace the faceplate without disturbing the filter, and fasten securely. Undertightening will allow air leakage, overtightening will damage the sponge-rubber faceplate gasket. A very light application of talcum powder may be used on the sponge-rubber faceplate gasket to prevent the filter from sticking. During inclement weather the sampler may be removed to a protected area for filter change. Close the roof of the shelter, run the sampler for about 5 minutes, connect the rotameter to the nipple on the back of the sampler, and read the rotameter ball with rotameter in a vertical position. Estimate to the nearest whole number. If the ball is fluctuating rapidly, tip the rotameter and slowly straighten it until the ball gives a constant reading. Disconnect the rotameter from the nipple; record the initial rotameter reading and the starting time and date on the filter folder. (The rotameter should never be connected to the sampler except when the flow is being measured.) Sample for 24 hours from midnight to midnight and take a final rotameter reading. Record the final rotameter reading and ending time and date on the filter folder. Remove the faceplate as described above and carefully remove the filter from the holder, touching only the outer edges. Fold the filter lengthwise so that only surfaces with collected particulates are in contact, and place in a manila folder. Record on the folder the filter number, location, and any other factors, such as meteorological conditions or razing of nearby buildings, that might affect the results. If the sample is defective, void it at this time. In order to obtain a valid sample, the high-volume sampler must be operated with the same rotameter and tubing that were used during its calibration.

7.2 *Analysis*. Equilibrate the exposed filters for 24 hours in the filter conditioning environment, then reweigh. After they are weighed, the filters may be saved for detailed chemical analysis.

7.3 *Maintenance*.

7.3.1 *Sampler Motor*. Replace brushes before they are worn to the point where motor damage can occur.

7.3.2 *Faceplate Gasket*. Replace when the margins of samples are no longer sharp. The gasket may be sealed to the faceplate with rubber cement or double-sided adhesive tape.

7.3.3 *Rotameter*. Clean as required, using alcohol.

8. *Calibration*.

8.1 *Purpose*. Since only a small portion of the total air sampled passes through the rotameter during measurement, the rotameter must be calibrated against actual airflow with the orifice calibration unit. Before the orifice calibration unit can be used to calibrate the rotameter, the orifice calibration unit itself must be calibrated against the positive displacement primary standard.

8.1.1 *Orifice Calibration Unit*. Attach the orifice calibration unit to the intake end of the positive displacement primary standard and attach a high-volume motor blower unit to the exhaust end of the primary standard. Connect one end of a differential manometer to the differential pressure tap of the orifice calibration unit and leave the other end open to the atmosphere. Operate the high-volume motor blower unit so that a series of different, but constant, airflows (usually six) are obtained for definite time periods. Record the reading on the differential manometer at each airflow. The different constant airflows are obtained by placing a

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series of loadplates, one at a time, between the calibration unit and the primary standard. Placing the orifice before the inlet reduces the pressure at the inlet of the primary standard below atmospheric; therefore, a correction must be made for the increase in volume caused by this decreased inlet pressure. Attach one end of a second differential manometer to an inlet pressure tap of the primary standard and leave the other open to the atmosphere. During each of the constant airflow measurements made above, measure the true inlet pressure of the primary standard with this second differential manometer. Measure atmospheric pressure and temperature. Correct the measured air volume to true air volume as directed in 9.1.1, then obtain true airflow rate, Q, as directed in 9.1.3. Plot the differential manometer readings of the orifice unit versus Q.

8.1.2 *High-Volume Sampler.* Assemble a high-volume sampler with a clean filter in place and run for at least 5 minutes. Attach a rotameter, read the ball, adjust so that the ball reads 65, and seal the adjusting mechanism so that it cannot be changed easily. Shut off motor, remove the filter, and attach the orifice calibration unit in its place. Operate the high-volume sampler at a series of different, but constant, airflows (usually six). Record the reading of the differential manometer on the orifice calibration unit, and record the readings of the rotameter at each flow. Measure atmospheric pressure and temperature. Convert the differential manometer reading to m.^{3}/min., Q, then plot rotameter reading versus Q.

8.1.3 *Correction for Differences in Pressure or Temperature.* See Addendum B.

9. *Calculations.*

9.1 *Calibration of Orifice.*

9.1.1 *True Air Volume.* Calculate the air volume measured by the positive displacement primary standard.

$$V_a = \frac{(P_b - P_m)}{P_b}(V_M)$$

V_a = True air volume at atmospheric pressure, m.^{3}
P_b = Barometric pressure, mm. Hg.
P_m = Pressure drop at inlet of primary standard, mm. Hg.
V_M = Volume measured by primary standard, m.^{3}

9.1.2 *Conversion Factors.*
Inches Hg. × 25.4 = mm. Hg.
Inches water × 73.48 × 10^{-3} = inches Hg.
Cubic feet air × 0.0284 = cubic meters air.

9.1.3 *True Airflow Rate.*

$$Q = \frac{V_a}{T}$$

Q = Flow rate, m.^{3}/min.
T = Time of flow, min.

9.2 *Sample Volume.*

9.2.1 *Volume Conversion.* Convert the initial and final rotameter readings to true airflow rate, Q, using calibration curve of 8.1.2.

9.2.2 *Calculate volume of air sampled*

$$* \quad V = \frac{Q_i Q_f}{2} \times T$$

V = Air volume sampled, m.^{3}
Q_i = Initial airflow rate, m.^{3}/min.
Q_f = Final airflow rate, m.^{3}/min.
T = Sampling time, min.

9.3 *Calculate mass concentration of suspended particulates*

$$S.P. = \frac{(W_f - W_i) \times 10^6}{V}$$

S.P. = Mass concentration of suspended particulates, μg/m.^{3}
W_i = Initial weight of filter, g.
W_f = Final weight of filter, g.
V = Air volume sampled, m.^{3}
10^6 = Conversion of g. to μg.

10. *References.*

(*1*) Robson, C. D., and Foster, K. E., "Evaluation of Air Particulate Sampling Equipment", *Am. Ind. Hyg. Assoc. J. 24,* 404 (1962).

(*2*) Tierney, G. P., and Conner, W. D., "Hygroscopic Effects on Weight Determinations of Particulates Collected on Glass-Fiber Filters", *Am. Ind. Hyg. Assoc. J. 28,* 363 (1967).

(*3*) Unpublished data based on a collaborative test involving 12 participants, conducted under the direction of the Methods Standardization Services Section of the National Air Pollution Control Administration, October, 1970.

(*4*) Harrison, W. K., Nader, J. S., and Fugman, F. S., "Constant Flow Regulators for High-Volume Air Sampler", *Am. Ind. Hyg. Assoc. J. 21,* 114–120 (1960).

(*5*) Pate, J. B., and Tabor, E. C., "Analytical Aspects of the Use of Glass-Fiber Filters for the Collection and Analysis of Atmospheric Particulate Matter", *Am. Ind. Hyg. Assoc. J. 23,* 144–130 (1962).

ADDENDA

A. *Alternative Equipment.*

A modification of the high-volume sampler incorporating a method for recording the actual airflow over the entire sampling period has been described, and is acceptable for measuring the concentration of suspended particulates (Henderson, J. S., Eighth Conference on Methods in Air Pollution and Industrial Hygiene Studies, 1967, Oakland,

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*This equation should read $V = \frac{(Q_i + Q_f)}{2} \times T$

Calif.). This modification consists of an exhaust orifice meter assembly connected through a transducer to a system for continuously recording airflow on a circular chart. The volume of air sampled is calculated by the following equation:

$$V = Q \times T.$$

Q = Average sampling rate, m.³/min.
T = Sampling time, minutes.

The average sampling rate, Q, is determined from the recorder chart by estimation if the flow rate does not vary more than 0.11 m.³/min. (4 ft.³/min.) during the sampling period. If the flow rate does vary more than 0.11 m.³ (4 ft.³/min.) during the sampling period, read the flow rate from the chart at 2-hour intervals and take the average.

B. *Pressure and Temperature Corrections.*

If the pressure or temperature during high-volume sampler calibration is substantially different from the pressure or temperature during orifice calibration, a correction of the flow rate, Q, may be required. If the pressures differ by no more than 15 percent and the temperatures differ by no more than 100 percent (°C), the error in the uncorrected flow rate will be no more than 15 percent. If necessary, obtain the corrected flow rate as directed below. This correction applies only to orifice meters having a constant orifice coefficient. The coefficient for the calibrating orifice described in 5.1.4 has been shown experimentally to be constant over the normal operating range of the high-volume sampler (0.6 to 2.2 m.³/min.; 20 to 78 ft.³/min.). Calculate corrected flow rate:

$$Q_2 = Q_1 \left[\frac{T_2 P_1}{T_1 P_2}\right]^{1/2}$$

Q_2 = Corrected flow rate, m.³/min.
Q_1 = Flow rate during high-volume sampler calibration (Section 8.1.2), m.³/min.
T_1 = Absolute temperature during orifice unit calibration (Section 8.1.1), °K or °R.
P_1 = Barometric pressure during orifice unit calibration (Section 8.1.1), mm. Hg.
T_2 = Absolute temperature during high-volume sampler calibration (Section 8.1.2), °K or °R.
P_2 = Barometric pressure during high-volume sampler calibration (Section 8.1.2), mm. Hg.

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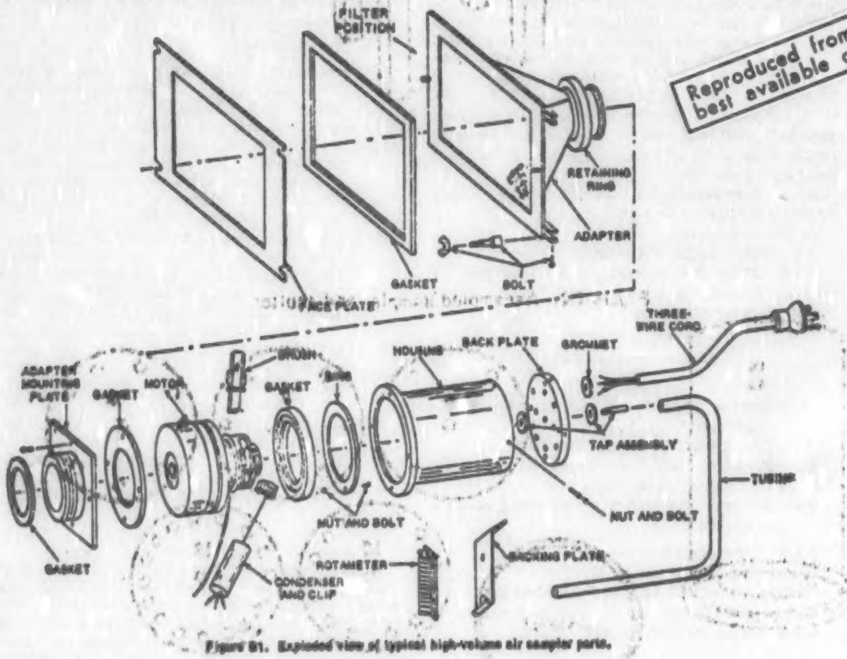
FILTER POSITION
RETAINING RING
ADAPTER
GASKET
BOLT
FACE PLATE
THREE-WIRE CORD
ADAPTER MOUNTING PLATE
GASKET
MOTOR
BRUSH
GASKET
RING
HOUSING
BACK PLATE
GROMMET
TAP ASSEMBLY
TUBING
NUT AND BOLT
NUT AND BOLT
GASKET
CONDENSER AND CLIP
ROTAMETER
BACKING PLATE

Figure B1. Exploded view of typical high-volume air sampler parts.

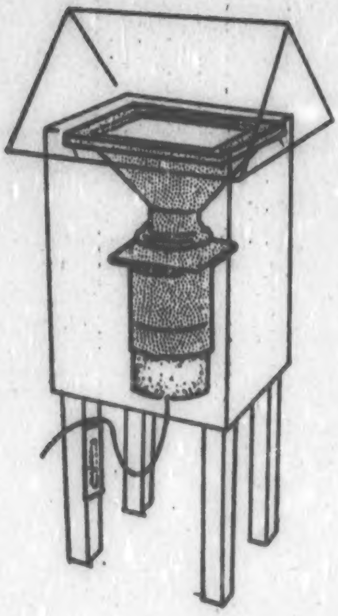

Figure B2. Assembled sampler and shelter.

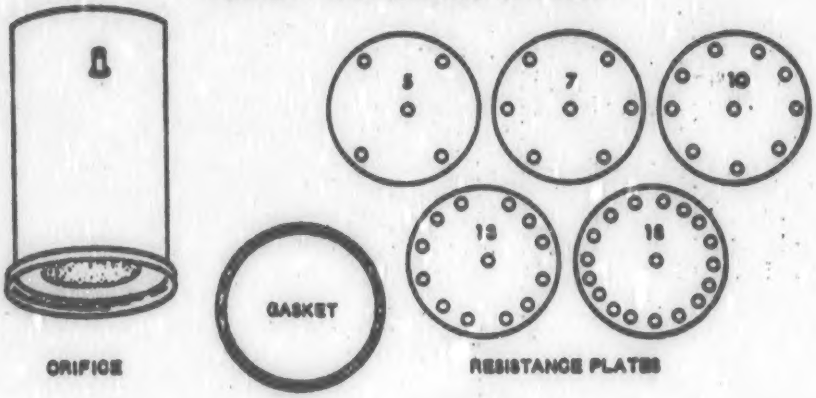
5
7
10
13
18
GASKET
ORIFICE
RESISTANCE PLATES

Figure B3. Orifice calibration unit.

11.0 REFERENCES

1. McKee, H. C., et.al. Collaborative Study of Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method). PB 205-891. June 1971.
2. Smith, F. and A. C. Nelson, Jr. Guidelines for Development of a Quality Assurance Program, Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method). EPA-R4-73-028b. June 1973.
3. The Monitoring and Field Activity of the National Air Surveillance Network, Standard Operating Procedures for EPA, Region IV. August 5, 1975.
4. Quality Assurance Handbook for Air Pollution Measurement Systems - Volume I, Principles. EPA-600/9-76-005. March 1976.
5. Cameron, J. M. Traceability? Journal of Quality Technology. 7(4):193-195, October 1975.

12.0 DATA FORMS

Blank data forms are provided on the following pages for the convenience of the Handbook user. Many of these forms are taken or adapted from EPA forms, Reference 2, and other references. No documentation is given on these forms as it would detract from their usefulness. The titles are also placed at the top of the figure as is customary for a data form. In order to relate the form to the test, a form number is given in the lower right-hand corner, e.g.,

Form TSP-2.4,

which implies that the form is Figure 2.4 in Section 2 of the TSP Handbook. Future revisions of these forms, if any, can be documented by 2.4A, 2.4B, etc. The data forms included are listed below.

Form	Title
1.1	Procurement Log
2.3	Timer Calibration Log
2.6	Hi Vol Orifice Calibration Log
2.10	Hi Vol Calibration Log
3.1	Laboratory Log for Total Suspended Particulate Data
3.2	Hi Vol Field Data Sheet
8.1	$\overline{X}$ and R Chart

PROCUREMENT LOG

Item description	Qty	Pur-chase order No.	Vendor	Date		Cost	Dispo-sition	Comments
				Ord	Rec			

TIMER CALIBRATION LOG

Site location	Timer No.	Date		Elapsed time indicator serial No.	Test period	Elapsed time indicator reading	Accuracy	By (signature)
		Start	Stop					

HI VOL ORIFICE CALIBRATION LOG

Description Derivation	Ft^3 of Air	m^3 of Air	Barometric Pressure mm Hg	Vacuum in Standard in mm Hg	Absolute Volume m^3	Time in Minutes	Flowrate m^3/min	Pressure Drop Across Orifice in IN of water
					$V_m(P_a-P_m)/P_a$		V_a/t	
Symbol / Plate Number	V_c	V_m	P_a	P_m	V_a	t	Q_1	P_t
	100	2.83						
	100	2.83						
	100	2.83						
	100	2.83						
	100	2.83						
	100	2.83						
	100	2.83						
	100	2.83						
	100	2.83						
	100	2.83						

Orifice Number__________ Manufacturer__________

Temperature_____ C Barometric Pressure________ Time________ EST

Date__________ Primary Standard__________ Serial Number_____

Calibration voltage__________ Signed__________

Verified By__________

Definitions for use with Figure 2.6

V_a = Actual volume of air measured in cubic meters.

V_c = Volume measured by the positive displacement meter in cubic feet

V_m = Volume measured by the positvie displacement meter in cubic meters as calculated from Vc.

P_a = Atmospheric pressure in mm Hg.

P_m = Vacuum at the inlet of the positive displacement meter in mm Hg.

t = Minutes of time elapsed during run.

Q_1 = Flow rate in cubic meters per minute at prevailing atmospheric pressure and temperature (uncorrected).

P_t = Pressure drop across the orifice in inches of water.

Equations given in Figure 2.6

$$V_m = V_c \times 0.0283$$

$$V_a = V_m \frac{(P_a - P_m)}{P_a}$$

$$Q_1 = \frac{V_a}{t}$$

HI VOL CALIBRATION LOG

Rotameter Number ______
Motor Number ______
Site Name ______

Description / Derivation / Plate Number	Initial				Final			
	Recorder or Rotameter Reading	Pressure drop Across the Orifice in inches of water	m^3/minute Flowrate Q1	Corrected Flowrate Q_2*	Recorder or Rotameter Reading	Pressure drop Across the Orifice in inches of water	m^3/minute Flowrate Q_1	Corrected Flowrate Q_2

Date ______ Time ______ Temperature ______
Barometric Pressure ______ Orifice Number ______ Voltage ______

Signed ______ Verified By ______

Date ______ Time ______ Temperature ______
Barometric Pressure ______ Orifice No. ______ Voltage ______

Signed ______ Verified By ______

$$^{*}Q_2 = Q_1\left(\frac{T_2T_1}{T_1P_2}\right)^{1/2}$$

LABORATORY LOG FOR TOTAL SUSPENDED PARTICULATE DATA

CITY NAME ____________________

SITE ADDRESS ____________________

PROJECT ________________ TIME INTERVAL

STATE ☐☐ AREA ☐☐☐☐ SITE ☐☐☐

PROJECT ☐☐ TIME ☐ YEAR ☐☐

INSTRUMENT LAST CALIBRATED ____________________

SAMPLER IDENTIFICATION NO. ____________________

TOTAL SUSPENDED PARTICULATE

NAME PARAMETER CODE

1 1 1 0 1

SAMPLING DATE MONTH	DAY	ST HR *	CONC µg/m³	FILTER NUMBER	AVERAGE * CFM	SAMPLING TIME	AIR m³ VOLUME	GROSS WGT.	TARE WGT.	NET WGT.	REMARKS

*FROM AVERAGE FLOW DETERMINED FROM CALIBRATION

LABORATORY FUNCTIONS

FILTER PREPARATION

- FILTER MUST BE FREE OF PINHOLES, TEARS, CREASES, LUMPS, ETC.
- FILTER MUST BE PROPERLY EQUILIBRATED (20 hrs < TIME < 28 hrs; HUMIDITY < 50%) (Δ HUMIDITY ± 5%; Δ TEMP ± 3°C)
- BALANCE MUST BE CHECKED BEFORE WEIGHING (STD. WGT. 3 to 5 g ± 0.0005 g)

SAMPLING ANALYSIS

- SAMPLE MUST BE FREE OF EVIDENCE OF MALFUNCTION; TEN OR FEWER INSECTS IN SAMPLE
- FILTER MUST BE PROPERLY EQUILIBRATED (SEE ABOVE)
- BALANCE MUST BE CHECKED BEFORE WEIGHING (SEE ABOVE)

ALL SAMPLES WERE HANDLED IN ACCORDANCE WITH THE ABOVE GUIDELINES

____
SIGNATURE

HI VOL FIELD DATA SHEET

HIGH-VOL DATA RECORD

Net Particulate Wgt.________________ grams

Air Volume________________ m^3

Particulate Concentration________________ $\mu g/m^3$

Total Sampling Time ________ hours ________ minutes

REMARKS________________

____

____

____

____

BACK

HI-VOL DATA RECORD

STATION LOCATION
CITY & STATE ________________

SITE ADDRESS________________

PROJECT________ SITE NO.________

INSTRUMENT LAST CALIBRATED________________

SAMPLER IDENTIFICATION NO.________________

FILTER NUMBER________________

START SAMPLING________________
mo day yr hr min CFM

STOP SAMPLING________________
mo day yr hr min CFM

WIND:____ calm, ____ light, ____ gusty

VISIBILITY:____ clear, ____ hazy

SKY:____ clear, ____ scattered, ____ overcast

HUMIDITY:____ dry, ____ moderate, ____ humid, ____

TEMPERATURE °F:____ <20, ____ 20-40, ____ 41-60, ____ 61-80 ____ >80

- Faceplate must be hand tight
- Flow rate must be ± 10% of established flow rate
- Faceplate gasket must be in good condition
- Rotameter must be free of foreign material
- Rotameter operation must be stable
- Sampler motor brushes must be changed every 400 hrs of operation

Sample was collected in accordance with the above guidelines

____
signature

REMARKS________________

____

____

____

FRONT

Quality Assurance Handbook TSP-3.2

$\bar{X}$ and R CHART

PROJECT NAME ____________ MEASUREMENT PERFORMED ____________ MEASUREMENT UNITS ____________

DATE																											
MEASUREMENT	CODE	1																									
		2																									
		3																									
	RESULT	1																									
		2																									
		3																									
SUM																											
AVERAGE, $\bar{X}$																											
RANGE, R